TEXTILE RESEARCH JOURNAL

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VOLUME 31 DECEMBER 1961 NUMBER 12

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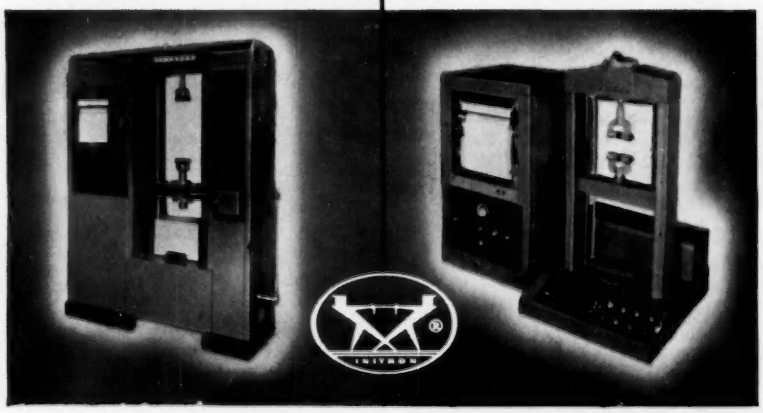

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TEXTILE RESEARCH JOURNAL

A Modification of the Method for Defining and Measuring Crimp of Textile Fibers[1]

M. Shiloh, J. Goldstein, D. Mejzler, and E. Alexander

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Abstract

The main parameter used to describe the geometry of the crimp of a fiber is the *crimp diameter*. This corresponds to an average amplitude of the fiber in space while the fiber is kept in a static position of stress.

In order to measure the crimp diameter, it is necessary to measure the coordinates of a sufficient number of points on the fibers. From these measurements, a "fiber axis" can be defined as the straight line for which the sum of squares, S, of the distances of the fiber points from this line is minimal. The crimp diameter is then defined as $D = 2\sqrt{S/N}$, where N is the number of points measured along the fiber.

The calculation of S according to this definition involves the solution of third-order equations. Therefore a simplified equation is suggested. It was found that this equation can, for all practical purposes, serve as a satisfactory approximation for the results obtained when S is calculated according to the definition, i.e., from the fiber axis.

In order to measure the crimp diameter, a special apparatus had to be built. A series of limitations of the rotatory crimp apparatus led to the modification of the measuring method and to the development of the method of two perpendicular planes. This method was applied to various types of fiber, such as cotton and fine wools.

Introduction

In a previous work [1], a method of defining the crimp of textile fibers was described. The following parameters were suggested as describing the geometry of the crimp of a fiber while it is kept in a static position of stress.

The main parameter used was the *crimp diameter*. This corresponds to the average amplitude of the fiber in space. In order to measure the crimp diameter it is necessary to measure the coordinates of a sufficient number of points on the fibers; from these the crimp diameter can be calculated.

Another parameter, the *wave number*, was taken as a second independent crimp parameter. This parameter can be obtained from the above defined crimp diameter and the stress–strain data of the crimped fiber, in the form of the *crimp ratio*. For the computations of the wave numbers two regular shaped models were used: a circular helix and a sine curve.

Additional information on the crimp can be obtained from the measurements of the stress–strain

[1] This work is a part of a Ph.D. thesis by M. Shiloh, submitted to the Hebrew University, Jerusalem, Israel.

behavior. Measurements of this behavior and the crimp ratio of the fiber may be carried out in various instruments, e.g., the Instron tester. However, in order to measure the crimp diameter a special apparatus is necessary.

The Rotatory Method

The first method used [1] involved a rotatory crimp apparatus and will therefore be referred to as the rotatory method.

In that method the fiber was rotated around an axis passing through its end points. Its image was projected on a screen. During the rotation each point of the fiber describes a circle in the plane perpendicular to the axis of rotation. In the projection, this circular motion will appear as a movement along a straight line, with the "width" of $2r_iM$, where r_i is the distance of the fiber point from the axis of rotation and M the magnification of the projection. Measurements of $2r_i$ were taken at N points at constant distances along the rotation axis. The crimp diameter is then calculated as

$$D = \left(\sum_{i=1}^{N} \frac{(2r_i)^2}{N} \right)^{\frac{1}{2}} \tag{1}$$

The line connecting the end points of the fiber is used in this method as a fiber axis from which the distances r_i of the fiber points should be measured. Actually, only the distances from the axis of rotation can be measured in this method. Therefore, much care has to be taken in bringing the end points of the fiber exactly on the rotation axis, and inaccuracies in this adjustment will influence seriously the resulting calculated crimp diameter.

In order to facilitate and speed up this adjustment of the end points on the rotation axis, the apparatus was further developed and improved. This was described in a subsequent paper [2]. However, the measurements were still too time consuming, especially considering the large number of measurements which are necessary in order to represent various samples of crimped fibers. The jaws had to be recentered each time they were opened for the application of additional loads to the fibers. A small hook had to be tied to one end of each fiber, on which additional loads were to be applied. All this required much time. Furthermore, the procedure of centering the jaws was not always successfully completed in practice: the movements of the motor and gears caused slight vibrations so that some surface of revolution of the axis was always present and could not be completely avoided. These inaccuracies had a negligible effect on the measurements of the wool and jute fibers used in the earlier work, but had some influence on the measurements of cotton and fine wool samples which were carried out later on.

Also, the illumination of the fiber in this instrument was too poor for cotton fibers, as the light was passing through the two mirrors; a higher intensity of illumination would have heated the fibers too much.

Another disadvantage of the rotatory method is the error which is caused by the random choice of fiber end points. When the fiber is inserted into the jaws, the point of contact is at a random phase of the wave; this point is used to define the axis of rotation around which the $2r_i$ values are determined. If, on the other hand, some other (random) point is chosen, the axis will generally be a different one; other width values might then be measured and a different value of D be obtained.

When all these limitations were considered, it seemed that an improvement of the method was necessary if fibers with smaller crimp diameters, such as cotton, were to be investigated. Therefore the following method of two perpendicular planes was developed.

Theoretical Considerations

The definition of the crimp diameter given earlier depends on the fiber end points which are chosen for the definition of the axis of measurement.

The following definition of the crimp diameter avoids this ambiguity. As the fiber axis from which the distances r_i are to be calculated, we define that straight line for which the sum of squares of the distances of the fiber points from it will be *minimal*. Let S be this minimal sum of squares; D will be now defined as

$$D = 2(S/N)^{\frac{1}{2}} \tag{2}$$

D describes approximately the volume occupied by the fiber in space, i.e., the diameter of an "average cylinder" which is defined by the fiber.

The line passing through the end points of the fiber (which is kept in a static state) is chosen as the axis of measurement. The coordinates x_i and z_i at N points of the fiber projection are measured. z is the coordinate along the axis of measurement and x is perpendicular to z.

A corresponding set of measurements (y, z) is taken in a second plane perpendicular to the plane (x, z). All distances $z_i - z_{i+1}$ are chosen to be equal.

These measurements are performed by imposing the fiber pictures on millimetric paper in such a way that z is coincident with one coordinate of the millimetric paper, as shown in Figure 1.

The Calculation of S

According to its definition, the calculation of S involves a least-squares analysis, which is complicated because three dimensions are involved.

For the calculation of S let us choose a set of points $P_i(x_i, y_i, z_i)$, let L be any straight line, and let us define the sum

$$S_L = \sum_{i=1}^{N} r_{iL}^2 \tag{3}$$

where r_{iL} is the distance of the point P_i from the line L. The value of S_L depends upon the position chosen for the line L. S is defined as the minimum of S_L. Any straight line is given by

$$(x - a)/m = (y - b)/n = (z - c)/p \tag{4}$$

All measurements are taken with loaded fibers, the unloaded state being unsuitable for practical measurements. The r_i are always small compared to the distance between the fiber end points. Therefore, it can be assumed that the minimal axis (fiber axis) will not be parallel to the (x, y) plane and therefore we can choose in Equation 4 $c = 0$ and look upon the group of lines which fulfill

$$(x - a)/m = (y - b)/n = z/p \quad \text{where } p \neq 0. \tag{5}$$

We can always choose

$$m^2 + n^2 + p^2 = 1 \tag{6}$$

when m, n, p are the cosines of direction of the line L. For the sum S_L we shall then get

$$S_L = \sum_{i=1}^{N} \{[p(y_i - b) - nz_i]^2 + [mz_i - p(x_i - a)]^2 + [n(z_i - a) - m(y_i - b)]^2\} \tag{7}$$

S_L depends on the parameters a, b, m, n, p which are also linked to the condition in Equation 6. It is now necessary to find those values of these parameters which will give a minimum of S_L.

In order to find the stationary points of the function $S(a, b, m, n, p)$ we shall use the method of

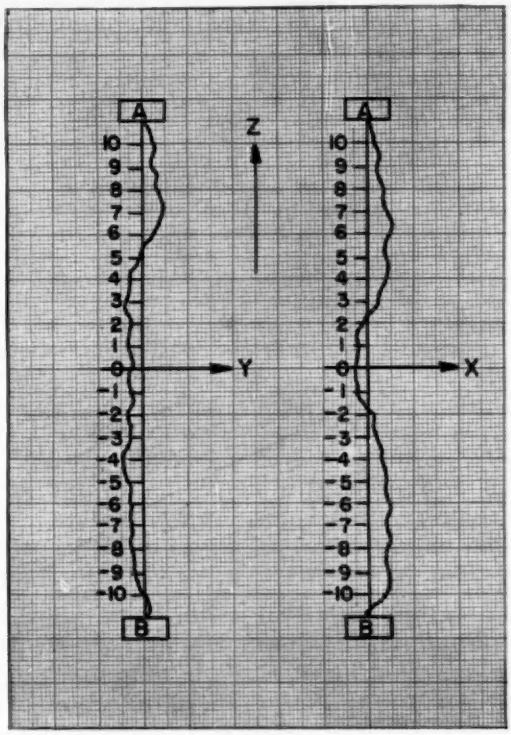


Fig. 1. The method of reading the coordinates of the points of the fiber.

Lagrange's multipliers. We define the function F

$$F(a, b, m, n, p) = S(a, b, m, n, p) - \lambda(m^2 + n^2 + p^2 - 1) \tag{8}$$

together with the condition in Equation 6. The minimum of F will also be the minimum of S_L. From $\partial F/\partial a = \partial F/\partial b = 0$ we get

$$a = \frac{p\sum x_i - m\sum z_i}{Np} \qquad b = \frac{p\sum y_i - n\sum z_i}{Np} \tag{9}$$

In order to get simpler equations for m, n, p we now choose new coordinates (also called x_i, y_i, z_i), so that the origin will be the center of gravity of the system of the N points, or

$$\sum x_i = \sum y_i = \sum z_i = 0 \tag{10}$$

so that

$$a = b = 0 \tag{11}$$

With this assumption we get as a necessary condition of minimum

$$\partial F/\partial m = \partial F/\partial n = \partial F/\partial p = 0 \tag{12}$$

and from Equation 12 the following equations for m, n, p are obtained

$$m[\sum(z_i^2 + y_i^2) - \lambda] + n(-\sum x_i y_i) + p(-\sum x_i z_i) = 0$$

$$m(-\sum x_i y_i) + n[\sum(z_i^2 + x_i^2) - \lambda] + p(-\sum y_i z_i) = 0$$

$$m(-\sum x_i z_i) + n(-\sum z_i y_i) + p[\sum(y_i^2 + x_i^2) - \lambda] = 0 \quad (13)$$

These form a set of homogeneous equations which have a nontrivial solution (other than $m = n = p = 0$) only in the case when the determinant of the coefficients is equal to zero, or

$$\Delta = \begin{vmatrix} \sum(z_i^2 + y_i^2) - \lambda & -\sum x_i y_i & -\sum x_i z_i \\ -\sum x_i y_i & \sum(z_i^2 + x_i^2) - \lambda & -\sum y_i z_i \\ -\sum x_i z_i & -\sum z_i y_i & \sum(y_i^2 + x_i^2) - \lambda \end{vmatrix} = 0 \quad (14)$$

The equation $\Delta = 0$ is the well-known secular equation. Because of the symmetry of the determinant, it is known that there are always three real solutions, λ_1, λ_2, λ_3. For each λ_k ($k = 1, 2, 3$) we get from Equations 13 together with the condition in Equation 6, a solution for (m_k, n_k, p_k). Therefore, there will always be three solution lines which will give the sums S_1, S_2, S_3. The necessary S will be the minimum:

$$S = \min(S_1, S_2, S_3) \quad (15)$$

It can also easily be seen that when Equations 13 are multiplied by m, n, and p respectively and summed together, we get $S = \lambda$; the solutions λ_k therefore give S directly, so that it is not necessary to calculate m, n, and p.

The Approximated Calculation of S

The actual calculation of S which involves the solution of a third-order equation in each case, is too laborious for practical purposes, considering the large number of calculations which are necessary in order to represent various fiber samples. It was therefore decided to calculate S through an explicit equation which may be referred to as an approximated equation.

Let us assume (and this assumption can be well justified by various experiments on crimped fibers) that the minimal axis (fiber axis) and the axis of measurement, z, are intersected an an angle φ which is small, so that

$$r_i = d_i \cos\varphi \simeq d_i$$

where r_i are the distances of the points $P_i(x_i, y_i, z_i)$ to the fiber axis and d_i are the projections of r_i on the (x, y) plane, as shown in Figure 2. Instead of the sum

$$S_L = \sum_{i=1}^{N} r_{iL}^2 \quad (3)$$

we shall now calculate

$$S_L \simeq S_L' = \sum_{i=1}^{N} d_{iL}^2 \quad (16)$$

Assuming that $p \neq 0$ we can now describe the line L by

$$(x - a)/m = (y - b)/n = z/1 \quad (17)$$

or

$$x = mz + a$$
$$y = nz + b \quad (18)$$

It is easily seen that

$$d_{iL}^2 = (mz_i + a - x_i)^2 + (nz_i + b - y_i)^2 \quad (19)$$

and the sum

$$S_L = S(m, n, a, b) = \sum d_{iL}^2 = \sum[(mz_i + a - x_i)^2 + (nz_i + b - y_i)^2] \quad (20)$$

m, n, a, b can be obtained from the necessary conditions of minimum for S_L

$$\begin{aligned} \tfrac{1}{2}\partial S_L/\partial m &= m\sum z_i^2 + a\sum z_i - \sum x_i z_i = 0 \\ \tfrac{1}{2}\partial S_L/\partial n &= n\sum z_i^2 + b\sum z_i - \sum y_i z_i = 0 \\ \tfrac{1}{2}\partial S_L/\partial a &= Na + m\sum z_i - \sum x_i = 0 \\ \tfrac{1}{2}\partial S_L/\partial b &= Nb + n\sum z_i - \sum y_i = 0 \end{aligned} \quad (21)$$

From Equation 21 we get

$$m = \frac{N\sum x_i z_i - \sum x_i \sum z_i}{N\sum z_i^2 - (\sum z_i)^2} = \frac{\sum(x_i - \bar{x})(z_i - \bar{z})}{\sum(z_i - \bar{z})^2}$$

$$n = \frac{N\sum y_i z_i - \sum y_i \sum z_i}{N\sum z_i^2 - (\sum z_i)^2} = \frac{\sum(y_i - \bar{y})(z_i - \bar{z})}{\sum(z_i - \bar{z})^2}$$

$$a = \bar{x} - m\bar{z} \qquad b = \bar{y} - n\bar{z} \quad (22)$$

It is therefore seen that the projection of the fiber axis (minimal axis) on the (x, z) plane, $x = mz + a$, is the regression line of the projections of the N given points on the (x, z) plane, and that

$y = nz + b$ is the corresponding regression line in the (y, z) plane.

From Equation 22 we get the following expression for S

$$S = \sum(x_i - \bar{x})^2 + \sum(y_i - \bar{y})^2 - \left[\frac{[\sum(x_i - \bar{x})(z_i - \bar{z})]^2 + [\sum(y_i - \bar{y})(z_i - \bar{z})]^2}{\sum(z_i - \bar{z})^2}\right] \quad (23)$$

or, using the notation used in statistics, we get

$$S = \text{var}(x) + \text{var}(y) - \left[\frac{[\text{cov}^2(x,z) + \text{cov}^2(y,z)]}{\text{var}(z)}\right] \quad (24)$$

In the actual measurements it is always possible to choose $\sum z_i = 0$ (as shown in Figure 1) so that the equation can be further simplified

$$S = \sum(x_i - \bar{x})^2 + \sum(y_i - \bar{y})^2 - \frac{[(\sum x_i z_i)^2 + (\sum y_i z_i)^2]}{\sum z_i^2} \quad (25)$$

and usually $\sum z_i^2$ may be constant for a certain series of measurements.

In these equations S can always be divided into two individual sums: $S = S_x + S_y$. These sums can be calculated from the (x, z) and (y, z) planes separately.

S is then introduced to Equation 2 in order to get the crimp diameter

$$D = 2(S/N)^{\frac{1}{2}} \quad (2)$$

A Numerical Example

The difference between the sum S and the approximated sum was calculated from the measured coordinates of various fibers and under various stresses. One of the most outstanding differences was obtained in the following case.

The coordinates x and y (in mm.) were measured from two perpendicular projections of a cotton fiber loaded with 2.5 mg. and with an enlargement of 31× (Table I). The value of S is found to be $1103.4/31^2$ (mm.^{2}) and $D = 0.479$ mm. The value of S obtained by the approximated equation is $1110.9/31^2$ (mm.^{2}) and $D = 0.481$ mm. It is seen that the difference does not exceed 0.7%, so that it is negligible when compared to the desired accuracy of the measurements.

The use of the approximated equation for S is therefore justified.

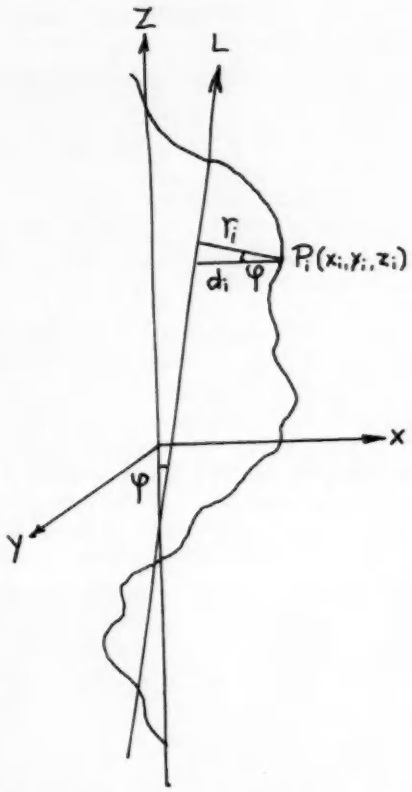


Fig. 2. The fiber axis and the axis of measurement.

TABLE I.

z	x	y
10	16.8	−14.8
9	4.2	8.0
8	3.0	−11.1
7	4.0	−9.8
6	10.0	−1.3
5	10.3	3.0
4	3.0	−1.2
3	−0.5	0.4
2	5.5	5.1
1	10.1	5.0
−1	−1.5	−0.6
−2	−2.7	−1.1
−3	2.0	−2.5
−4	−2.3	6.0
−5	9.0	−9.0
−6	3.4	−8.3
−7	1.5	−12.3
−8	0.6	−5.8
−9	0	−4.0
−10	0	5.4

Experimental

The New Crimp Meter

According to the new definition of the fiber axis, it is not necessary to rotate the fiber; instead of rotation we get two perpendicular projections of two planes of the fiber.

The measurements of the extensions and the crimp ratio are carried out in the same manner as in the rotatory apparatus, but the crimp diameter is obtained from the measurements on each plane separately.

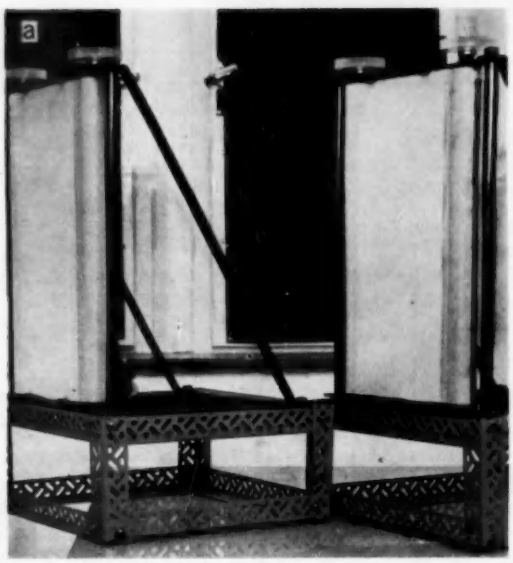
Let the z axis be the straight line which passes through the end points on each plane. These end points are the only marked points along the fiber and therefore allow the simultaneous choice of the z axis on both planes. Each point $P_i(x_i, y_i, z_i)$ of the fiber will describe the points (x_i, z_i) and (y_i, z_i) on both projection planes. From these projected points the minimal sums of squares, S_x and S_y, may be calculated, and the crimp diameter may be obtained

$$D = 2(S/N)^{\frac{1}{2}} = 2(S_x/N + S_y/N)^{\frac{1}{2}}$$

by using Equations 23 or 24.

The projections can be obtained on screens at various distances from the fiber and at various enlargements. Figure 3 shows the screens which were used in the present work on cotton fibers, at a distance of 3 m. from the fiber with an enlargement of 20×. In order to save space, one projection was directed through a mirror at 45° to the beam of light, so that both projections were obtained on one table. The mirror is also shown in Figure 4.

In Figure 4 the apparatus which has been built for this method of measurement is shown. In this apparatus the upper fiber end is fastened to a small jaw which is attached to a steel rod. This rod is hooked to a torsion balance. The lower end of the fiber is inserted into a jaw which is connected to a moving table. The movement of the table which carries the jaw is controlled by the lower handle, which is shown in the figure. Two lenses are rigidly connected to this table on two perpendicular arms. At the back part of the instrument, opposite to the arms, two sodium discharge lamps illuminate the fiber so as to cause the formation of the images of two of its planes through the lenses on the screens. To the moving table (which can be



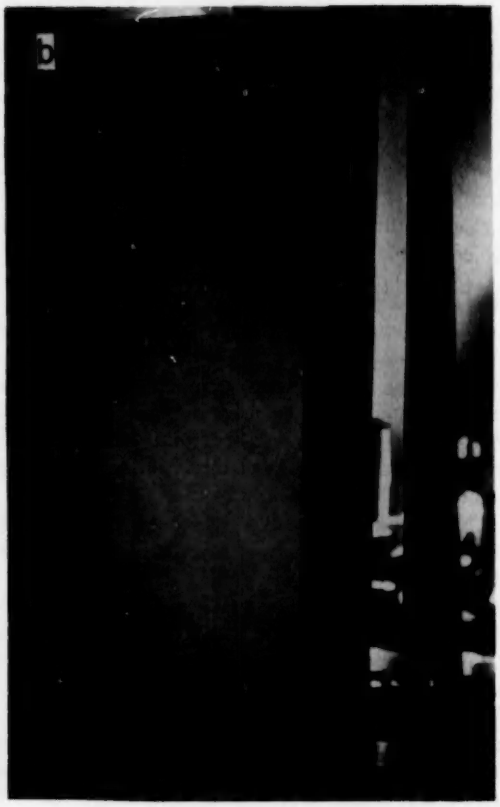


Fig. 3. The projection screens: a, a general view; b, the projection of the fiber on one plane.

lifted or lowered with the aid of the screw under the apparatus, as shown in Figure 4) a millimetric gauge is connected; on this gauge, the movement of the table, which corresponds to the stretching of the fiber, can be read and controlled. It is possible to counterweight the torsion balance ("Hartman and Braun," full scale reading of 2500 mg., maximum load 25 g.) against the weight of the rod and the jaw, on which the upper end of the fiber is attached. When the fiber is stretched, the weight which is necessary to lift the rod can be measured on the balance; this weight is equivalent to the load which caused the stretching of the fiber. In this way the static stress–strain characteristics of the fiber can be obtained, starting at 5 mg. and ending at breakage.

This method of mounting and measuring proved far more satisfactory than the previous method where weights had to be attached to the fibers and the extensions measured by a comparator.

The Mounting of the Fibers

A special method of gluing the fibers has been developed, in order to reduce handling of the fibers and to simplify the measurements on the crimp meter. Celluloid sheets, of a thickness of 0.5 mm., have been cut in the form shown in Figure 5.

The fibers are glued at the points A and B by an organic solution which dissolves the celluloid in the neighborhood of the points of contact. In this way slippage of fibers in the jaws can be avoided.

This frame is later cut (at the dotted lines C, D) into individual frames. For the measurement, a single frame is hung on the upper jaw of the crimp meter, the lower jaw of the apparatus is then closed on the lower end, B, of the frame. Now the vertical sides of the frame are cut away. From this position the fiber can be subjected to crimp measurements at various controlled extensions.

This method of fastening the fibers is also suitable for the tests of these and other fibers on the Instron tester, in order to get their stress–strain

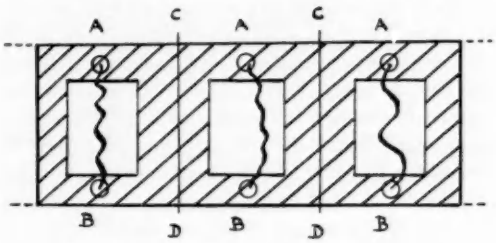


Fig. 5. The mounting of the fibers; fibers are glued to the frame.

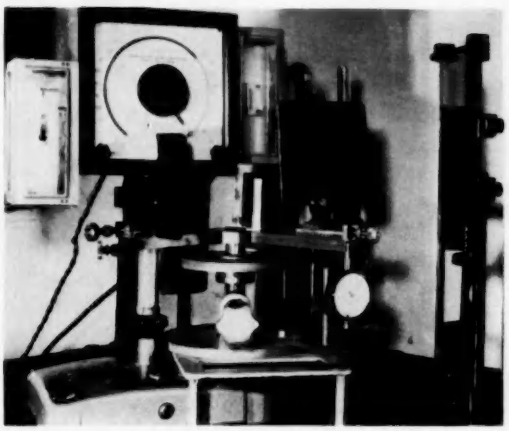


Fig. 4. The crimp meter.

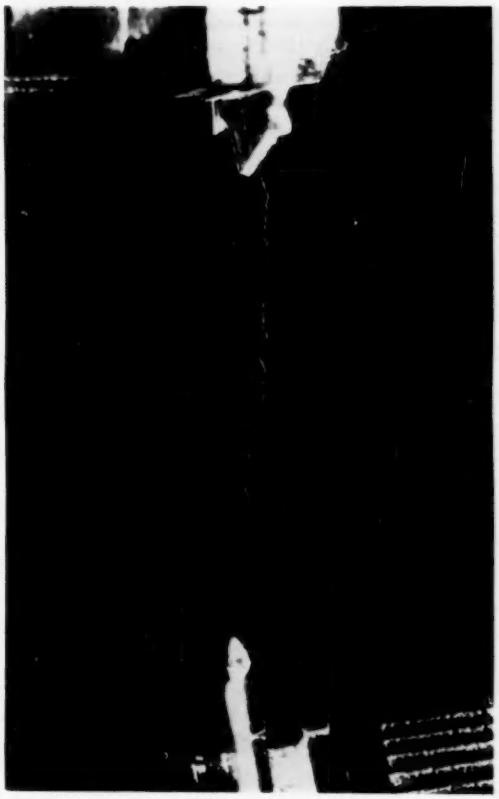


Fig. 6. A crimped cotton fiber (5×).

continuous curves and their uncrimping energies.[2] The method of fastening the fibers to the frame can be observed in Figure 4. Figure 6 shows a cotton fiber which was glued to such a frame and photographed on the Instron tester.

The Procedure of the Measurement

After the fiber has been inserted into the crimp meter, the lenses are set up for the appropriate magnifications of the projections on the screens. The points A and B, where the fiber is glued to the frame, are easily recognized on the screens because of the transparency of the celluloid; these points are therefore chosen as the ones defining the axis of measurement, z, as they ensure the same axis on both (x, z) and (y, z) planes, and there are no other marked points along the fiber.

Transparent drawing paper is used in order to copy the projection images on both screens. These drawings are later used in order to read the the coordinates; each drawing is put upon millimetric paper in the way shown in Figure 1. The end points A and B are directed so as to be on top of a marked line of the millimetric paper which will serve as the z axis. A line which is perpendicular to this line at the center of the portion AB is defined as the x or y axis. Then the deviations x_i or y_i from the z axis are read at N constant intervals of z; these values are later introduced into Equations 23, 24, or the simplified Equation 25. From this S and therefore D are obtained.

[2] The comparison of the results obtained for the uncrimping energies with the data of D for the same fibers will be given in a subsequent work.

Conclusions

The method of two perpendicular planes of a fiber has proved to be a simplified and more accurate method of measuring the crimp diameter of various fibers.

The use of the approximated equation for the minimal sum of squares of the distances of the fiber points to the fiber axis is justified for all practical purposes.

The same equations can also be used to measure the sum of squares of the deviations of any set of points from their regression line.

Acknowledgment

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A part of this work, which involved measurements of crimp of wool fibers, was supported by a grant given by the International Wool Secretariat, London.

The authors wish to express their thanks to Dr. M. Lewin, Director of Research of this Institute, for his assistance in carrying out this work.

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Boundary Friction of Textile Yarns

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Abstract

A method and apparatus for determining the boundary friction of textile fibers are described. Mineral oil and oleic acid are shown to be ineffective boundary lubricants for "Dacron," [1] nylon, acetate, rayon, and polyacrylonitrile fibers. Stearic acid and *n*-octadecylamine reduced the boundary friction compared to that of the clean fibers. Polyacrylic acid grafted to nylon gave low boundary friction when treated with *n*-octadecylamine. Oleic acid formed an effective boundary film on chrome which gave low friction when rubbed by various textile fibers and was stable at temperatures up to 160° C. Lubricants had no effect on the boundary friction of "Teflon." [2] These results are interpreted in terms of adsorption of the lubricants on the rubbing surfaces.

Introduction

From observations made on various textile processing operations, it is obvious that fiber surface boundary friction plays an important role in product processability and quality. For example, while ropes require low boundary friction for maximum strength, it cannot be categorically stated that low boundary friction is always best. Therefore, it is essential to know which factors influence the boundary friction of textile fibers in order to understand yarn processing characteristics and product properties. Some of these factors are (1) the adsorption of chemical compounds by the fiber or guide surface, (2) the roughness of the surfaces, and (3) the rheological properties of the lubricant. This investigation was concerned with some effects of adsorption on the boundary lubrication of textile fibers.

Experimental Procedure

The apparatus used in this study consisted of a slow-speed drive which pulled a strand of yarn over a fixed surface at 0.01 cm./sec. The free end of the strand was loaded with a 30-g. weight, and the tensions were recorded using a Brown recorder and a Statham strain gauge. A schematic diagram is shown in Figure 1. The boundary coefficient of friction was calculated from the force required to maintain movement of the strand over the test item. All measurements, unless otherwise stated, were made at 22° C. and 65% RH. Friction coefficients were calculated using the belt equation, $T_2/T_1 = e^{f\theta}$, where T_1 and T_2 are the input and output tensions, f is the friction coefficient, and θ is the wrap angle in radians.

Yarn-to-yarn friction measurements were made on ¼-in.-diameter steel cylinders wrapped with a complete cover of the test yarn with a helix angle of about 15°. Friction for yarn-to-guide materials was determined against ¼-in. polished cylinders. The effect of temperature on friction was measured on a ½-in. polished chrome sleeve containing a ¼-in.-diameter "Chromalox" [3] heater cartridge. An "Amplitrol" [4] control unit equipped with an iron-constantan thermocouple was used to regulate the temperature.

Results

Boundary Friction of Clean Fibers

Boundary frictional forces are greatest when the two surfaces rubbed together are clean. Preparation of a clean surface is often difficult. However, if cleaning is continued until the friction reaches a maximum and several different cleaning procedures give the same maximum frictional value, the fiber surfaces may be considered free of contaminants. Another approach is to assume that friction between sliding surfaces is caused by welds formed at the

[1] Registered trademark for Du Pont's polyester fiber.
[2] Registered trademark for Du Pont's TFE-fluorocarbon fiber.
[3] Manufactured by the E. L. Wiegand Co.
[4] Made by the Thermo Electric Manufacturing Co., Dubuque, Iowa.

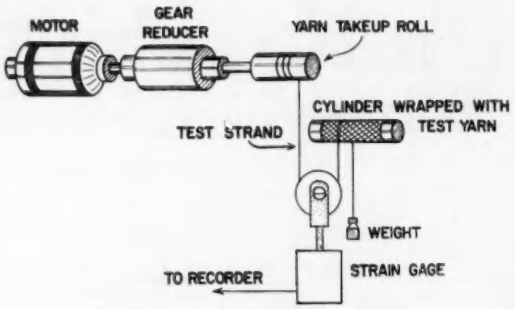

Fig. 1. Boundary friction apparatus.

points of contact (asperities) and that subsequent shearing occurs within the bulk of the polymer, not at the interface. If these conditions hold, the friction coefficient is equal to the polymer's bulk shear strength divided by its yield pressure, and boundary lubricants function by interposing a layer of low shear strength material between the sliding surfaces [3]. The shear strength/yield pressure ratio is given by Zisman and co-workers [4] as 0.6 for nylon, while Chapman, Pascoe, and Tabor give values of 0.63 and 0.33, respectively, for polyethylene and Teflon at 20° C. [5]. Friction coefficients of 0.1 or less have been reported for Teflon, and these results are discussed by Allan [1] who believes a different mechanism of sliding operates with this polymer.

The procedure used in this study for removing lubricants from fibers consisted of alternate 1-ml. rinses of methanol and 1,1,1-trichloroethane ("Chlorothene," Dow Chemical Co.). The fiber and guide surfaces were cleaned in place on the friction apparatus. The results showed that carefully cleaned samples of fibers produced by Du Pont gave boundary coefficients which ranged from 0.45 to 0.8, with the exception of Teflon for which the value was 0.10. The various fibers are arranged in order of increasing coefficients of friction in Figure 2.

Effect of Mineral Oil

Theoretically, since pure mineral oil contains no polar groups, it cannot chemically bond itself to fiber surfaces and, when present as a lubricant, should not reduce the boundary coefficient of friction appreciably compared to that of the clean fibers. However, it was observed in every case that the boundary friction was slightly lowered when No. 50 mineral oil ("Bayol" 50, Esso Standard Oil Co., Charlotte, N. C.) was flooded between the clean fiber faces. It is suspected that some of these long-chain lubricant molecules are physically adsorbed to the polymer surface in a flattened orientation. Data are shown in Figure 3.

Effect of Oleic Acid

Oleic acid contains the polar carboxyl group, which makes it a potentially good boundary lubricant for any surface to which this group will bond. On clean metal surfaces, oleic acid can form monolayers which produce boundary friction coefficients of 0.10 or less [3]. One would expect, if a similar monolayer were formed on fiber surfaces, that a low coefficient of boundary friction would be obtained. Experimental data showed, however, that oleic acid (Hormel Institute, Division of G. A. Hormel Co., Austin, Minn.) was still a relatively poor boundary lubricant on the fibers tested. Presumably, the polymer bonding sites are too far apart to allow the formation of a close packed monolayer, and any adsorption which occurred probably consisted of molecules lying flat on the yarn surface. The data are shown in Figure 4.

Effect of Stearic Acid

Stearic acid is a solid at 22° C. and should form a more effective lubricant film than oleic acid because of the stronger cohesive forces between adsorbed molecules. Boundary friction measurements showed that stearic acid (Distillation Products Industries, Rochester 3, N. Y., recrystallized from ethanol) was a more effective lubricant than oleic acid, as illus-

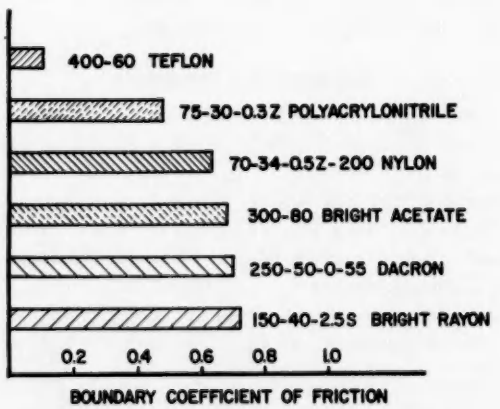

Fig. 2. Boundary friction between clean fibers.

trated in Figure 5. The friction of Teflon was not affected by this lubricant.

Effect of n-Octadecylamine

n-Octadeclyamine (Research Division, Armour & Co., Chicago, Ill.) is also a solid at 22° C. and contains the primary amine group as a potential bonding site for polymer surfaces. It reduced the boundary friction for these fibers to essentially the same level as stearic acid, except for rayon where the value was lower. Again, there was no effect in the case of Teflon. The data are shown in Figure 6.

Boundary Friction of Polyacrylic-Acid-Grafted Nylon

It was thought that nylon yarn, upon whose surface polyacrylic acid had been grafted, would offer a high probability of chemisorbing a boundary lubricant because of the large number of carboxyl groups exposed at the surface. A substance which should be adsorbed by the polyacrylic acid surface is *n*-octadecylamine. When this amine was applied to the grafted nylon surface from carbon tetrachloride solution, the friction coefficient immediately dropped to the range 0.07 to 0.09, compared to the range 0.45 to 0.50 for the clean surface. As expected, oleic acid and No. 50 mineral oil did not reduce the boundary friction appreciably, giving values of 0.45 to 0.49. When *n*-octadecylamine was applied to a regular 6-6 nylon surface (70-34-0.5Z-200), the coefficient found was 0.40, compared to 0.63 for the clean fiber. Therefore, it is concluded that *n*-octadecylamine is not strongly adsorbed by the regular nylon, and the low frictional value for the poly-

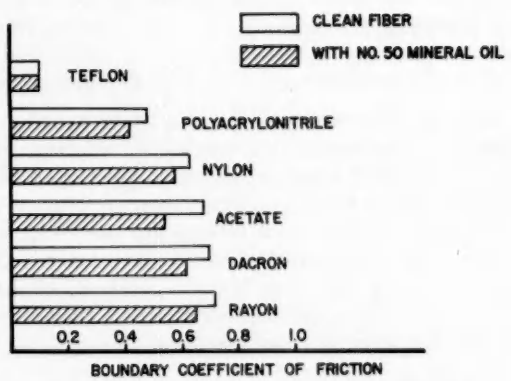

Fig. 3. Mineral oil as a boundary lubricant.

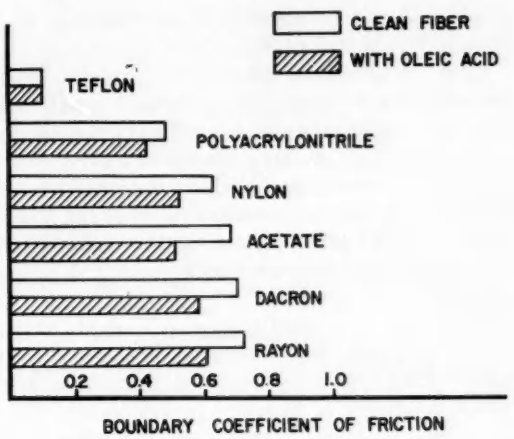

Fig. 4. Oleic acid as a boundary lubricant.

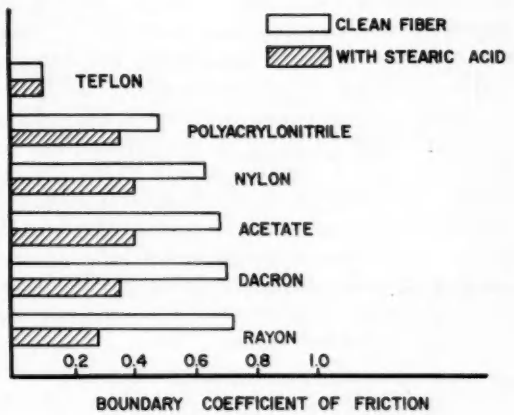

Fig. 5. Stearic acid as a boundary lubricant.

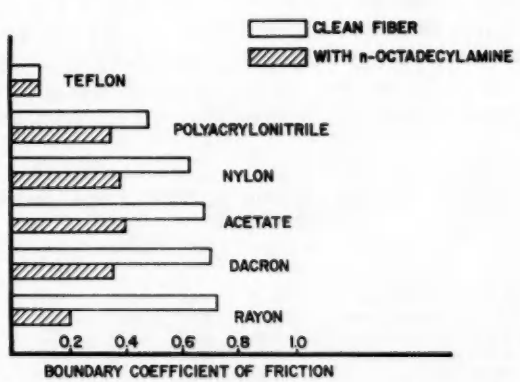

Fig. 6. *n*-Octadecylamine as a boundary lubricant.

acrylic-acid-grafted material is a reflection of a chemisorbed boundary film.

Close packed solid monolayers are quite strong and will support large loads; whereas, weakly held films are easily penetrated. Although the friction experiments indicate the formation of a close packed monolayer of *n*-octadecylamine on the grafted nylon, further tests such as resistance to repeated sliding over the same track and temperature stability should be made to determine the film strength and surface coverage [6].

The behavior of wool is another example of how surface properties can be changed by chemical treatment. Lindberg [7] pointed out that virgin wool, whose coefficient of boundary friction is low (0.1), is not wet by water. After treatment with caustic or ultraviolet radiation, the original low friction surface was destroyed, the wool became hydrophilic, and the coefficient rose to 0.3 or more, reflecting the appearance of a new and more polar surface. It is even possible to change the surface properties of Teflon by treating it with sodium and liquid ammonia [8]. Measurements by Allan [2] showed that the contact angle of water on this polymer was reduced from 108° (untreated surface) to 50° (treated surface) and that the boundary friction coefficient simultaneously rose from 0.15 to 0.4. The surface of the treated material became more polar and the forces of attraction much stronger so that the shearing of junctions during frictional measurements now took place in the bulk plastic.

Boundary Friction of Fibers on Other Solid Surfaces

The boundary friction of clean fibers against other clean solid surfaces should be independent of the kind of material, provided its shear strength is equal to or greater than that of the fiber and shearing does not take place at the junction interfaces [3]. Friction coefficents observed for clean fibers sliding over other solid surfaces support this concept. Data are listed in Table I and compare yarn-to-yarn friction with that of yarn against one-quarter inch diameter rods of chrome, "AlSiMag"[5] 586 (TiO_2), and synthetic sapphire (Al_2O_3), which are typical textile guide materials. Although the frictional values for the different substrates are not identical for any given fiber, they agree well enough to indicate that the boundary friction is controlled by the shear strength of the fiber, which is the weaker material. Teflon exhibits a low friction in every case; the shearing of junctions is undoubtedly taking place at the guide-to-ploymer interface [1].

TABLE I. Boundary Friction of Clean Fibers Against Different Substrates

	Yarn-to-yarn	Chrome	"AlSiMag" 586	Sapphire
70-34-0.5Z-200 Nylon	0.63	0.56	0.60	0.55
150-40-2.5S Bright rayon	0.72	0.80	0.81	0.70
300-80 Bright acetate	0.68	0.70	0.75	0.71
75-30-0.3Z Polyacrylonitrile	0.48	0.47	0.50	0.42
400-60 Teflon	0.10	0.10	0.10	0.10
250-50-0-5S Dacron	0.70	0.73	0.71	0.73

When lubricants are present, the chemical properties of the guide as well as those of the polymer surface become highly important in influencing boundary friction. The chemical aspects of the fiber surface have already been discussed, and metal surfaces will be considered next. It is known that clean metal surfaces can adsorb many boundary lubricants to form solid monolayers. Chrome plate was used in this study since it is an important surface in textile processing. The chromium surface was carefully cleaned and then flooded with oleic acid. The surface quickly became autophobic and the excess oleic acid contracted into small drops which exhibited a large contact angle. A clean strand of nylon or Dacron yarn rubbing against this surface gave a boundary coefficient of friction of 0.10, which is a characteristic value for a solid monolayer and shows that adsorption on only one of two rubbing surfaces is sufficient to give low boundary friction. Teflon yarn gave a boundary coefficient of 0.10 against clean chrome; this is the same as its fiber value (0.10). This showed that low boundary friction is an inherent property of this fiber surface and does not necessarily depend on the nature of the other rubbing surface.

Effect of Temperature on Boundary Friction

Oleic acid on chrome provided a strong boundary film which maintained low friction at temperatures up to about 160° C. when rubbed by either nylon or Dacron yarns. A typical curve is shown in Figure 7. The reason for the initial drop in friction (0.10 to 0.03) as the temperature increased is not understood at present, but apparently is not a pecularity of the oleic acid–chrome system, since Teflon showed a similar behavior on clean chrome.

[5] Registered trademark of American Lava Corporation.

Teflon has a surface whose boundary friction is inherently low, and its behavior over a range of temperatures should give a good indication of the stability of the surface to oxidation or decomposition. Its frictional properties against clean chrome showed that it maintained low boundary friction to a temperature of at least 260° C. The data are shown in Figure 8.

Summary

The fibers listed above, carefully cleaned, gave boundary friction coefficients of 0.45 or more when rubbed against either themselves or ceramic or metal surfaces, except for Teflon whose coefficient was 0.1. The boundary coefficient of friction could be lowered to 0.1 or less for all fibers with the establishment of an effective lubricating film between the rubbing surfaces. However, this excellent degree of boundary lubrication was obtainable only for fiber-to-metal contacts, where the boundary layer was present on the metal rather than on the fiber surface. For fiber-to-fiber contacts, only poor to fair boundary lubrication was provided for the basic fibers by any one of the lubricants evaluated (mineral oil, oleic acid, *n*-octadecylamine, and stearic acid).

However, effective boundary films can be produced on textile fibers as well as on metals if sufficient bonding sites are present. For example, the boundary coefficient of friction for a clean sample of 15 den. nylon grafted with polyacrylic acid was reduced from 0.5 to 0.1 by treatment with *n*-octadecylamine.

Clean Teflon yarn rubbed against itself or against clean chrome gave a boundary coefficient of only 0.1. The behavior of Teflon is atypical, with shearing appearing to take place at the interfacial contact points rather than in the bulk structure which is characteristic of most other polymers in the nonlubricated state. The lubricants tested had no effect on the friction of Teflon.

Temperatures above 60° C. lowered the boundary friction coefficient from 0.1 to about 0.03 for various fibers in contact with a chrome surface having a boundary layer of oleic acid and for clean Teflon against clean chrome.

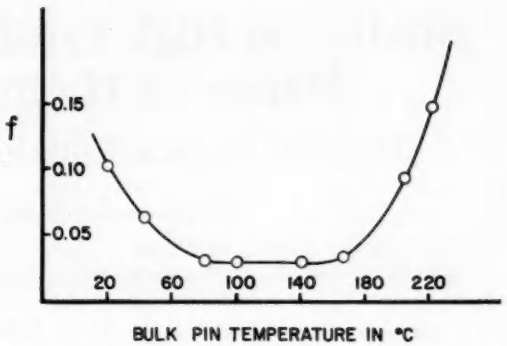

Fig. 7. Boundary friction of nylon vs. temperature on chrome lubricated with oleic acid.

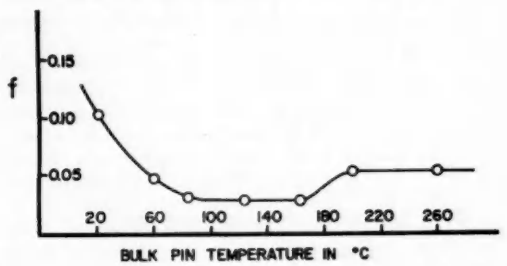

Fig. 8. Boundary friction of Teflon against clean chrome at different temperatures.

Acknowledgment

The authors wish to thank the E. I. du Pont de Nemours Company for permission to publish this paper.

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Studies on High Fastness to Light in Coloring Matters in Hydrophobic Substrates

Disperse Dyes in Cellulose Acetates and Polyesters

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Abstract

Disperse dyes C. I. 12770, 61100, and 61105, have been applied to disoriented polyester film, oriented polyester film and fiber (Terylene), and to cellulose secondary acetate and triacetate (films and fibers). Some control tests with a direct cotton dye (C. I. 24410) on regenerated cellulose films (normal and gel state) have been included. Spectral absorption (*x*- and *y*-band) and lightfastness (fading rate and CF curve) measurements have been made in dry and damp atmospheres.

The dyes become increasingly aggregated with rise in concentration, in each substrate, and also with rise in porosity (i.e., moisture regain) of all the substrates. The results show that if the adsorbed dye were freely accessible to water vapor and air, lightfastness would in all cases follow the normal rule and increase with the degree of aggregation of the dye, and thus with the moisture regain of the substrate. In substrates of very low regain, however, i.e., polyesters, fading is retarded by the "diffusion-restriction effect," i.e., the restricted access of, probably, water vapor and air to the dye particles. Lightfastness of disperse dyes on polyesters is thus higher than would be expected, and in one case (C. I. 61100) it is better on Terylene fiber than on cellulose acetates. In films lightfastness rises in the order: disoriented polyester < oriented polyester < cellulose triacetate < cellulose secondary acetate.

A hydrogen-bonding carrier (4-hydroxydiphenyl) reduces the degree of aggregation of a disperse dye in polyester film. This effect probably accounts for its deleterious effect on the lightfastness of disperse dyes on Terylene fiber.

The results agree with published data on lightfastness of disperse dye on cellulose acetate and polyester fibers.

In agreement with Color Index data, the present results appear to show that symmetry of substitution in anthraquinone disperse dyes favors the diffusion-restriction effect in polyester fibers and thus tends to raise lightfastness.

Introduction

Many dyes have much higher lightfastness on the hydrophobic fibers (i.e., those with a regain of < *ca.* 5%) than on fibers of higher regain. This applies particularly to a number of (a) disperse dyes, which have higher fastness rating on polyester fibers than on cellulose acetate; and (b) basic dyes, which have higher fastness on polyacrylonitrile fibers than on cotton, silk, or wool [4, 12]. This effect is the reverse of the general rule on the more hydrophilic fibers, on which the lightfastness rises with fiber regain. The fibers with higher regain have the larger intermolecular spaces, and consequently during absorption the dye molecules can apparently aggregate more readily in them. In general the larger the aggregates, the higher their lightfastness. This applies particularly to direct dyes on regenerated cellulose (viscose) of different degrees of crystallinity, either in fibrous or film form [2].

It was suggested [4] that on hydrophobic fibers the anomalously high fastness of many dyes is due to retardation of fading by the low rate of diffusion of moisture to the site of fading ("diffusion-restriction" effect).

The present investigation was made to examine this suggestion, for which no direct experimental evidence had previously been available. Three typical disperse dyes were chosen, one having higher (published) rated fastness on Terylene polyester fiber than on cellulose acetate fiber, and two the re-

verse (Table I). They were applied to polyester fibers and polyester and cellulose acetate films of different degrees of crystallinity. Also, some tests on regenerated cellulose films of different degrees of crystallinity were included for comparison. Spectral absorption curves, fading-rate curves, and characteristic-fading (CF) curves were determined. For full discussions of the significance of differences in these classes of curve see, e.g., [3, 8], [1, 7], and [2, 4, 6], respectively; shorter comments are given below.

Experimental

The regenerated cellulose films were Cellophane (0.001 in.) in two forms, the normal commercial quality and the gel state sheet, undried after manufacture.

Polyester fabric (Terylene, 4 den. fil.) and film (0.002 in., "Melinex") and cellulose (sec.) acetate fabric and film (0.001 in.), and cellulose triacetate fabric and film (0.003 in.) were used, all unplasticized. Disoriented polyester was made by dissolving Melinex film (0.8 g.) in boiling N-methyl-2-pyrrolidone (100 cm.^{3}), pouring, as hot as possible, onto glass plates, and drying by heat in still air to give a translucent film. Dyeings were made by the normal methods, at *ca.* 83° C. and at *ca.* 97° C. in open beakers for 90 min. for cellulose acetate, and cellulose triacetate, respectively, and at 120° C. for 60–75 min. in a domestic pressure cooker, for all the polyester materials, except one series of Melinex films (see Table I), which was dyed at boiling point in an open beaker in presence of *p*-phenylphenol as carrier (3% on wt. of film). The dyes were of normal commercial quality. Tested by alumina chromatography, C.I. 12770 and 61100 showed no colored impurities; C.I. 61105 had a considerable proportion of a redder component (probably the diamino compound, identical with C.I. 61100) and a trace of a greener component [probably the di(methylamino) derivative]. Acetone extracts from the C.I. 61105 dyed (CTA) films showed (apparently) a lower proportion of the redder component than in the original dye (and no greener material). The results for C.I. 61105 may be less significant than those for the other dyes.

The fading tests were made in mercury vapor light in the apparatus previously described and illustrated [7], either fully exposed to the air ("low humidity") or in stoppered tubes over water ("high humidity").

TABLE I. Details of Disperse Dyes and Hydrophobic Substrates Used

A. Dyes

C.I. No.	Name and constitution	Lightfastness [9, 10] (pale shade, no carrier)	
		Cellulose (sec.) acetate	Terylene polyester fiber
12770	Dispersol Yellow 3G (aniline → 2,4-quinoline diol)	3	1
61100	Duranol Violet 2R (1,4-diaminoanthraquinone)	4–5	5–6
61105	Duranol Brilliant Violet B (1, amino-4, methylaminoanthraquinone)	3–4	2

B. Substrates

Fiber or film	Moisture adsorption, % (approx., at 20° C.)
Terylene polyester fiber (4 den.) (T)	0.20
Melinex polyester film (0.002 in.) (OPE)	0.32
Recrystallized polyester film (0.002 in.) (PE)	0.97
Cellulose triacetate fiber (CTA)	3.9
Cellulose triacetate film (0.003 in.) (CTA)	4.5
Cellulose (sec.) acetate fiber (CA)	5.0
Cellulose (sec.) acetate film (0.001 in.) (CA)	5.5

TABLE II. Spectral Absorption Data for Dyes Adsorbed in Films

A. *Direct Cotton Dye (C.I. 24410, Chlorazol Sky Blue FF) in Regenerated Cellulose Films* *

Cellophane (normal film, 0.002 in.) (6050, 6600)†		Cellophane (gel film, 0.0025 in. after drying) (6050, 6550).	
(x)†	y/x†	(x)	y/x
0.294	0.80	0.196	0.82
0.502	0.80	0.387	0.84
0.820	0.80	0.500	0.85
1.004	0.83	0.773	0.87
1.600	0.87	0.989	0.90
		1.420	0.92
		1.800	0.92

B. *Disperse Dyes in Hydrophobic Films*

OPE		PE		CTA		CA	
(x)	y/x	(x)	y/x	(x)	y/x	(x)	y/x
C.I. 12770							
(4000, 4300)†		(4000, 4300)		(4000, 4275)		(4000, 4275)	
0.222	0.81	0.124	0.80	0.560	0.84	0.298	0.84
0.345	0.81	0.186	0.84			0.453	0.84
0.748	0.82	0.318	0.85			0.650	0.85
0.986	0.83	0.926	0.84	0.894	0.85	0.832	0.87
1.746	0.83	1.714	0.85	1.682	0.85	1.610	0.89
C.I. 61100							
(5550, 5900)		(5550, 5900)		(5450, 5900)		(5500, 5900)	
0.205	1.11	0.202	1.10	0.186	1.13	0.276	1.07
0.278	1.12	0.209	1.12			0.330	1.07
0.500	1.13	0.308	1.12			0.499	1.07
		0.316	1.12				
1.08	1.15	0.984	1.14	0.920	1.14	1.060	1.09
1.84	1.18	1.765	1.17	1.750	1.16	1.746	1.09
C.I. 61100 dyed with 4-hydroxydiphenyl carrier							
0.882	1.12						
1.020	1.14						
1.515	1.16						
C.I. 61105							
(5800, 6200)		(5800, 6200)		(5750, 6050)		(5750, 6050)	
0.128	1.00	0.189	1.04	0.204	1.14	0.179	1.15
0.241	1.00	0.175	1.05			0.256	1.14
0.416	1.04	0.303	1.04			0.253	1.24
						0.352	1.44
0.125	1.01	0.118	1.04	0.109	1.08	0.092	1.14
0.524	1.04	0.487	1.04	0.522	1.10	0.568	1.40
1.237	1.07	1.194	1.07	0.992	1.14	1.002	1.42
1.854	1.10	1.752	1.10	2.020	1.24	2.042	1.45

The moisture adsorption data (Table I) are mean values of duplicate measurements of the difference in equilibrated weight (of portions of *ca.* 0.5 gm.) at 110° C. and at room temperature in a stoppered tube over water.

A Unicam SP600 spectrophotometer was used, and readings of high-density films (> 1.0) were taken using a blank colored with a weaker shade of the same dye, for increased precision.

Discussion

State of Dye in the Substrate

The relative degrees of association of the dyes in the substrates may be judged qualitatively by comparison of spectra. Most dyes have two absorption bands in the visible spectrum: the short-wave (y) band usually increases in intensity more rapidly than the long-wave (x) band with increase in dye concentration, both in solution and in the adsorbed state, due to association of the dye molecules [8]. The y/x optical density ratio is therefore a qualitative measure of the degree of association. (It is assumed here that comparisons of y/x ratios *between* as well as *within* the different substrates are valid.)

The following conclusions are thus drawn from the present results (Table II, Figures 1, 2).

1. With one exception (see below), the degree of association of the dyes rises both with the porosity (i.e., the regain) of the substrate and with concentration. The effect is most marked with the direct dye in cellulose (Figure 2), and with the disperse dye (C.I. 61105) in cellulose acetates (Figure 3).

2. The monomer peaks of dyes C.I. 12770 and 61105 are in each case at higher wavelengths on polyester than on cellulose acetates. This indicates a closer alignment of dye and substrate molecules in the polyester than in cellulose acetate, leading to improved planarity and increased conjugation in the dye molecule. A similar shift of monomer waveband occurs when direct cotton dyes are transferred from water to cellulose [cf. 8], for the same reason.

* Lightfastness tests (Figures 14 and 16 of [2]) show that this dye is faster to light in the gel film than in the normal film.

† y and x waveband (λ_{max}) values (A) are shown in parenthesis; (x) = optical density of film at λ_{max} of x-band; y/x = ratio of optical densities at λ_{max} of y- and x-bands. Differences in optical density down to *ca* 0.01 unit are significant [8]. Duplicate sets of dyeings are grouped together.

Effect of Hydrogen-Bonding Carrier

A hydrogen-bonding carrier (4-hydroxydiphenyl) reduces the y/x band ratio of dye C.I. 61100 in polyester film (see Table II and Figure 3). Thus it is a disaggregating agent, as is phenol [8]. This is consistent with the conclusion reached from a study of published lightfastness data (Table III) which shows that this carrier has a marked effect in decreasing the lightfastness of disperse dyes on Terylene polyester fiber, whereas the corresponding non-hydrogen-bonding carrier, diphenyl, has no such effect. 2-Hydroxy-diphenyl, which for steric reasons is expected to be less effective as a hydrogen-bonding agent, has an intermediate effect. Phenol, in the dyebath, disaggregates a number of water-soluble dyes, and also reduces their lightfastness [1, 3, 8].

Anomalous Behavior of C.I. 61100

C.I. 61100 is anomalous in two respects; (i) the long-wave absorption peak is not at a higher wavelength in the dyed polyesters than in the cellulose acetates, and (ii) on dyed secondary cellulose acetate (not triacetate), the y/x ratio is much lower than on the other substrates. It is suggested that

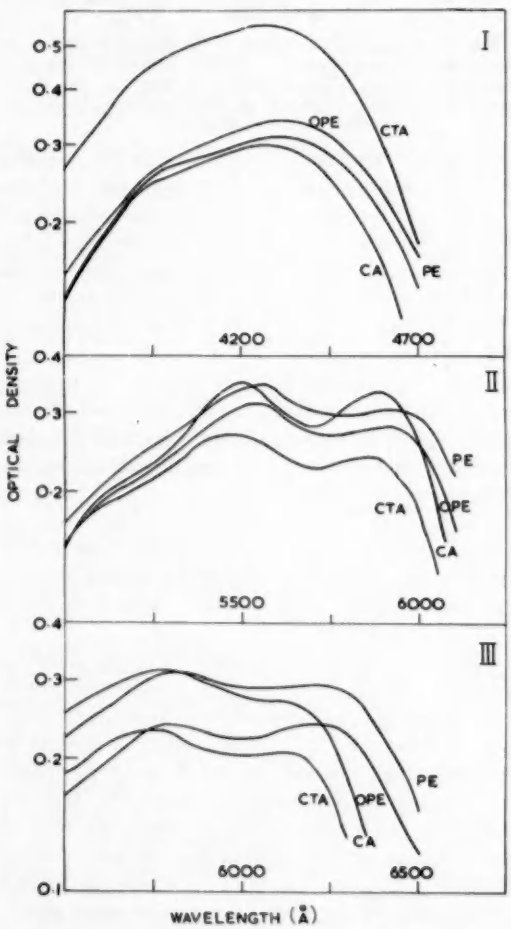

Fig. 1. Absorption spectra of disperse dyes in hydrophobic films. I, C.I. 12770; II, C.I. 61100; III, C.I. 61105. CA = cellulose (sec.) acetate; CTA = cellulose triacetate; OPE = oriented polyester; PE = disoriented polyester.

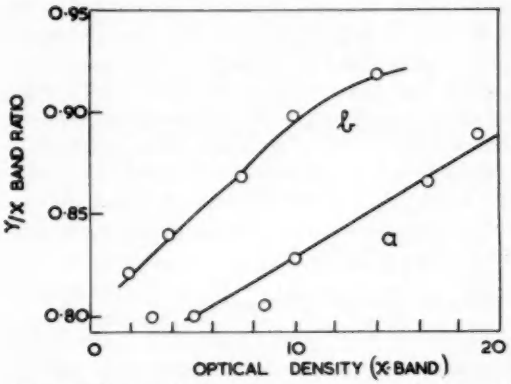

Fig. 2. Curves showing change in optical density ratio (y/x bands) with rise in concentration of C.I. Direct Blue I (24410) in regenerated cellulose films. (a) Normal film; (b) gel film. The corresponding CF curves (Figures 14, 16, of ref. [2]) show that the dye is about twice as fast to light in gel film as in normal film.

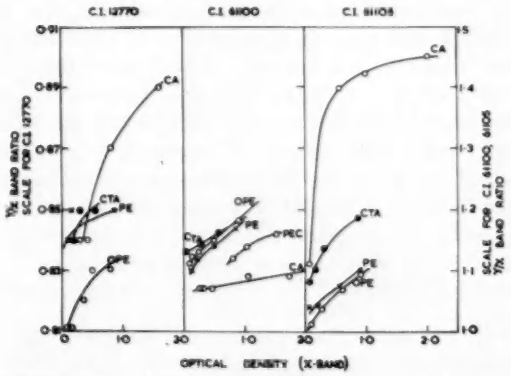

Fig. 3. Curves showing change in optical density ratio (y/x bands) with rise in concentration of disperse dyes in hydrophobic films. Optical densities from Table II, reduced to 0.001 in. film thickness. PEC = polyester film dyed at boiling point with carrier (3% 4-hydroxydiphenyl, on film weight).

TABLE III. Effect of Carriers in Reducing the Daylight Fastness of (Heavy Shades of) 20 Disperse Dyes on Terylene Polyester Fiber [10]

Carrier	Diphenyl	2-Hydroxy-diphenyl	4-Hydroxy-diphenyl
No. of dyes affected	0	11	14
No. unaffected	20	9	6
Mean reduction in fastness (grades)	—	0.9	1.3

the two 1 : 4-amino groups favor dye–dye association rather than dye–polyester association, thus accounting for effect (i), and cause a small amount of dye to be cross-linked to the hydroxy groups in secondary acetate, and to remain unassociated, thus accounting for effect (ii). There is in fact a rapid, but small, initial loss of dye in fading on secondary acetate (not observed with the other substrates or dyes) which is consistent with the presence of some monomer. This initial loss does not affect the position of the CF curves, which are calculated from later stages of fading.

Formation of Dye Aggregates

Temperature of dyeing does not influence the degree of aggregation. This seems to be the conclusion from the y/x ratios of C.I. 12770, where the polyesters, dyed at 120° C., give nearly as high ratios as the acetates, dyed at 85°–100° C. The dyeing mechanism of the present substrates is usually considered as a "solid solution" process induced by specific substrate–dye polar forces [5], but it appears that even so a proportion at least of the dye molecules associate after adsorption. There is evidence (see e.g. [3, 8]) that the dye associates at the substrate surface immediately it leaves the solution, which indeed would be expected, rather than on subsequent drying. The behavior of gel cellulose film also is consistent with this mechanism, the larger dye aggregates that form in it (Figure 2) must do so *before* drying, because when dried the material crystallizes [1] and becomes similar to normal film.

Lightfastness: the Diffusion-Restriction Effect

Fastness in disoriented polyester (PE), cellulose triacetate (CTA), and secondary cellulose acetate

[1] It does so also even when aged for a long period wet (Figures 16, 17 of ref. [2]).

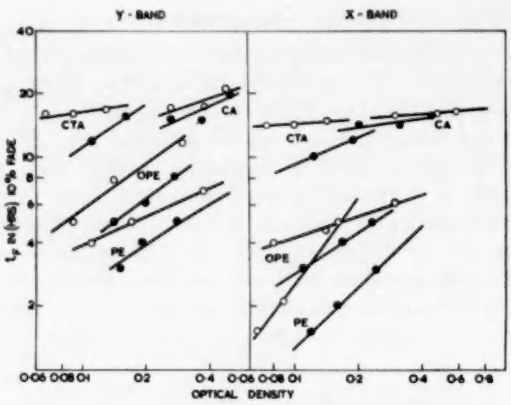

Fig. 4. Characteristic fading (CF) curves at y- and x-bands for disperse dye, C.I. 12770. Initial concentration figures are optical densities reduced to 0.001 in. film thickness. ○, normal atmosphere; ●, high-humidity atmosphere.

(CA) films increases in that order, i.e., in order of increasing regain and increasing aggregation of dye (Figures 4, 5, 6). Thus the normal rule is followed. But the rule is reversed by the other polyester substrates, with which fastness rises in the order: disoriented polyester < oriented polyester < Terylene fiber (Figures 5, 7). This is evidence of the diffusion-restriction effect. Even in a high-humidity atmosphere this reversal is still evident, and it seems likely therefore that some of the adsorbed dye is so embedded in a matrix of polyester molecules that it cannot be reached at all by the chemical agent causing fading.

All the tests show that fading is more rapid (a) under damp than under dry conditions,[2] and (b) at the x-band (monomer) than at the y-band (aggregate) wavelength. Both these effects occur with most other dyes [7].

The lightfastness of disperse dyes on Terylene polyester fiber is in most cases considerably lower when tested by Fade-Ometer than by daylight [10]. We are informed [11] that this effect is unlikely to be due to high humidity in the Fade-Ometer tests, because the effective humidity was 0–5%, but that it may be due to the excess of ultraviolet light in the Fade-Ometer arc light or to high temperature. Possibly sufficient UV light is absorbed by the phenyl nuclei of the fiber molecules to cause photolytic degradation and catalytic breakdown of dye.

[2] Except for C.I. 61100 on CA at the y-band wavelength; as mentioned above this dye on CA is anomalous in other respects also.

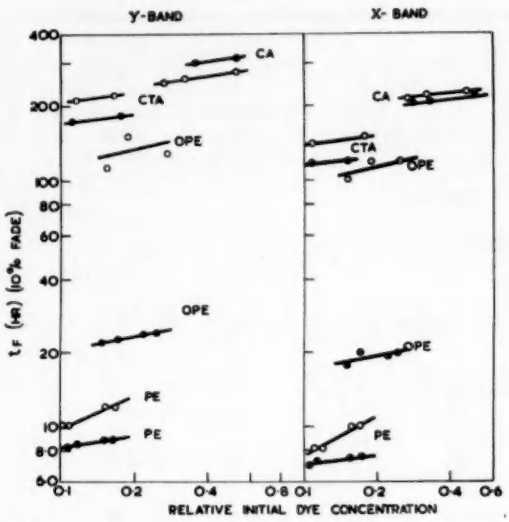

Fig. 5. CF curves at y- and x-bands for disperse dye C.I. 61100.

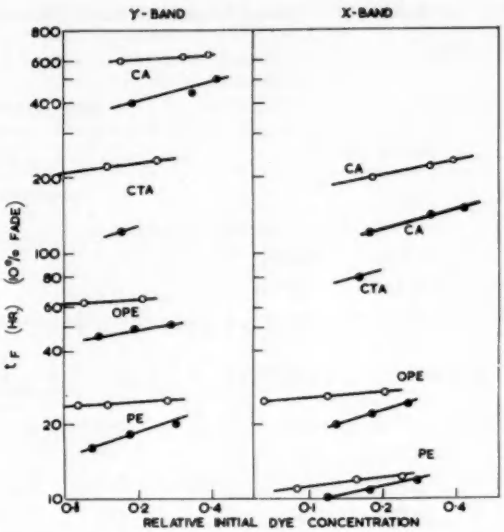

Fig. 6. CF curves at y- and x-bands for disperse dye C.I. 61105.

The higher fastness of C. I. 61100 on Terylene fiber, compared with cellulose acetate, agrees with published data (Table I). Inspection of the CF curves (Figures 4, 6) for the other dyes (not tested here on fibers) suggests that they would be less fast on Terylene than on cellulose acetate, which also agrees with the published data.

Relation between Lightfastness on Polyester Fiber and Dye Structure

When lightfastness data and chemical constitutions for anthraquinone disperse dyes are studied (Table IV) it is seen that symmetry of substitution in the 1, 4, 5, 8 positions favors high fastness on Terylene. It has already been pointed out [5] that symmetry of substitution in disperse dyes reduces their water solubility and their maximum adsorption by cellulose acetate, apparently by increasing the difficulty with which individual molecules can break away from the crystal structure to enter either the solution or the fiber. This increased stability of the crystal form is presumably the cause of the high fastness on Terylene; in some way it must favor blockage of the substrate pores by dye.

Fading-Rate Curves

These curves (cf. Figure 8) do not, except in one case (C.I. 61100 on CA, discussed above), show the initial sharp drop in density that occurs with many water-soluble dyes on hydrophilic fibers [1, 7] and is believed to be due to the presence of a small proportion of dye in the monodisperse state, readily accessible to water vapor and air. The shapes of the fading-rate curves are very similar for both monomer (x) and aggregate (y) peaks. This is attributed to the overlap of the two absorptions, i.e., the optical density at each peak is the sum of values for the two forms, the monomer form predominating at the higher wavelength and the aggregate form at the lower [cf. 3]. Thus it is not possible to obtain a

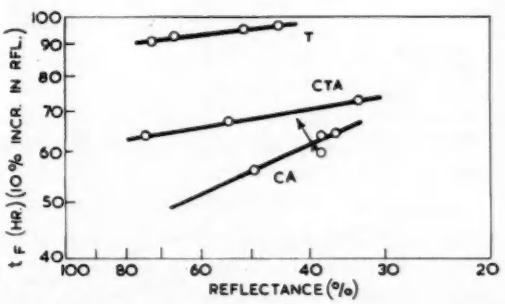

Fig. 7. CF curves (x-band) for disperse dye C.I. 61100 on fabrics. (T = Terylene.) The t_F values are for 10% gain in reflectance, and reflectance values have not been correlated with dye contents. On films (Figure 5) this dye is almost equally as fast on CTA as on CA; on fabrics it is significantly faster on CTA. The fiber has the lower regain (cf. Table I), thus there may be some diffusion-restriction effect (cf. p. 155 of [4]).

TABLE IV. Relation between Molecular Symmetry of Anthraquinone Disperse Dyes and Lightfastness

C.I. No.	Substituents 1	2	4	5	8	Lightfastness [9, 10] (Pale shade, no carrier) Cellulose (sec.) acetate	Polyester fiber (Terylene)
			Non-symmetrical compounds				
58900	(3-methoxybenzanthrone)					3–4	2
60505	$NHCH_3$					4–5	5
61105	$NHCH_3$		NH_2			3–4	2
61505	$NHCH_3$		NHC_2H_4OH			4	2
61515	$NHCH_3$		NHPh			4–5	3
			Symmetrical compounds				
60710	OH*		NH_2*			5–6	6
61100	NH_2		NH_2			4–5	5–6
62015	NH_2	OCH_3	NH_2			4–5	5
62500	OH		OH	NHC_2H_4OH	NHC_2H_4OH	4–5	4
63305	OH		$NHCH_3$	OH	$NHCH_3$	4	5

* These polar groups are similar in size and are here considered to represent "symmetrical" substituents.

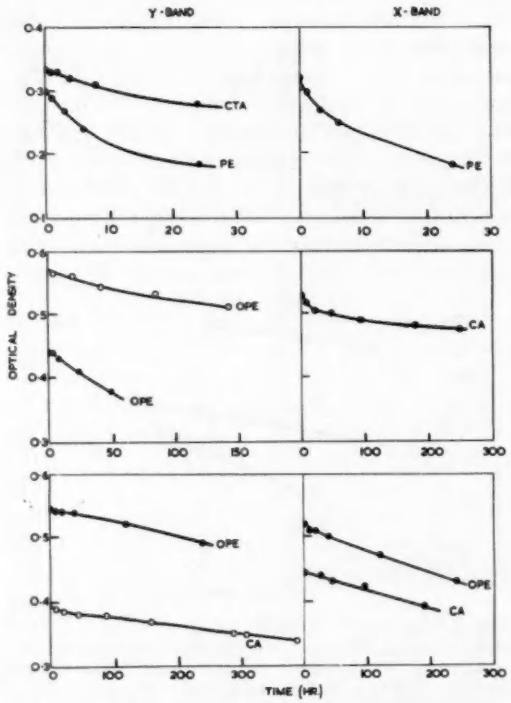

Fig. 8. Typical fading-rate curves for disperse dyes in hydrophobic films. Top, C.I. 12770; middle, C.I. 61100; bottom C.I. 61105.

fading curve characteristic of truly monomeric dye; in all cases the fading of a mixture of monomer and aggregate is measured.

Acknowledgments

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A Study of the Interaction between 2-Thiobarbituric Acid and Cotton

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Abstract

Solutions of 2-thiobarbituric acid have been shown to interact with raw cotton to form colored compounds. This interaction has been previously suggested as a test for radiation-damaged cotton [7]. Experiments were conducted to determine whether this interaction could be used as a diagnostic test for cotton which had been heat-damaged, as might occur during commercial ginning. While this test was found to be sensitive to the heating history of a given cotton, it was not possible to establish suitable absolute standards because of large differences among cottons. Investigation of the differences among cottons revealed that the interaction between 2-thiobarbituric acid and cotton is related to the alkali scourable matter in raw cotton as well as to fiber maturity. Possible uses for this test are suggested.

Introduction

Pan, Proctor, and Goldblith [7] have shown that solutions of 2-thiobarbituric acid (TBA) interact with radiation-damaged cotton to form a colored compound. The amount of colored compound formed, as determined by colorimetric intensity, was shown to be proportional to the radiation dose to which the cotton had been subjected. The authors suggested that the interaction between 2-thiobarbituric acid and radiation-exposed cotton be used as a means of estimating the damage due to the radiation, and that the test might be useful for estimating other forms of cotton damage as well.

2-Thiobarbituric acid and similar compounds, such as *p*-amino benzoic acid, have been extensively used in the field of biology for the analytical evaluation of degraded lipids [7]. The mechanism of the reaction between 2-thiobarbituric acid and degraded lipids to form colored compounds is not fully understood, and the active principle in degraded lipids which interacts with 2-thiobarbituric acid is not known. Most recent evidence indicates that the interaction is with an oxidized form of an unsaturated fatty acid [1].

The investigation reported in this paper was undertaken in order to elucidate further the nature of the interaction between 2-thiobarbituric acid and cotton and to explore the applicability of this interaction as a diagnostic test for heat damage in cotton.

Experimental

The experimental procedure is essentially that developed by Pan, Proctor, and Goldblith [7]. Reagent grade 2-thiobarbituric acid was dissolved in 50% acetic acid to make a 0.025 *M* solution (3.6 g./liter). In order to hasten wetting, 0.01% Ultrawet 30 DS was added to the solution. The reagent solution was found to be stable at room temperature for several days, but a fresh solution was prepared weekly. For the test, 250 mg. of cotton was weighed

on an analytical balance into a 150-mm. Pyrex test tube and 10 ml. of the reagent solution pipeted into the test tube. The cotton was dispersed in the reagent solution using a glass rod; the tube was then placed in a boiling water bath for exactly 15 min. and subsequently cooled in running cold tap water for 20 min. In most cases the cotton was cut into small pieces and, in the case of raw stock cotton, large pieces of foreign matter such as seed bits were removed by hand.

After the appropriate heating and cooling times, the supernatant solutions were decanted into silica cells designed for a Model DU Beckman spectrophotometer and the optical transmission at a wavelength of 449 mμ recorded. The reagent solution, without a cotton sample and treated in the same manner as the actual samples, was used as a blank for the spectrophotometer readings. The TBA value is defined as the transmission in percentage units.[1] On this basis, therefore, high TBA values (approaching 100) indicate no interaction between cotton and the solution of 2-thiobarbituric acid.

Effect of Heat

During the drying operation in commercial ginning, seed cotton is subjected to sufficiently high temperatures to cause either permanent, temporary, or latent effects on the fiber which reduce the spinnability and quality of the cotton [2, 3, 6]. A means of identifying heat-damaged cotton would, therefore, be extremely useful to the cotton textile industry.

In order to determine whether the TBA test is a sensitive measure of heat degradation, three samples of seed cotton were heated at 200° C. for varying lengths of time. Seed cotton was used in these experiments to be certain that the cottons had not previously been subjected to heat as is the case during commercial ginning. For these experiments, the fiber was removed from the seed by hand. The very strong dependence of the TBA value on the extent of heat treatment is shown in Figure 1. The initial heating appears to have the most marked effect, and since heating times below 2 min. are of particular interest in regard to damage during ginning it would seem that this test might be useful for diagnostic purposes. Also evident from Figure 1, however, is the fact that the three cottons have different TBA values in their original unheated conditions. Thus, a single TBA value for a cotton of unknown history cannot be used to determine whether a sample has been subjected to excessive heat. The differences in the original TBA values of the three cottons are not due to previous heat damage since seed cotton was used for these experiments. The differences in TBA values for different cottons must be explained by some other factor, a matter which will be considered in detail later.

A further attempt was made to adapt the TBA test as a diagnostic test for heat damage by noting the decrease in TBA value after cotton is subjected to a known increment of heat. Hopefully, it might be possible to establish the position of an unknown cotton on the time axis of Figure 1. Thus, if a cotton had been subjected to excessive heat prior to the test it might be located on the portion of the curve which is essentially level and the added increment of heat would cause only a minor decrease in TBA value. Conversely, a cotton which had not been subjected to excessive heat prior to the test would be positioned on the initial steep portion of the curve in Figure 1, and the known increment of heat would cause a large decrease in the TBA value. In order to establish the feasibility of this approach, it was necessary to have available samples of known heating history and to determine the change in TBA value of such samples after they had been subjected to a standardized increment of heat in the laboratory. For this purpose samples which constituted the 1957 California Gin Test were obtained from Joanna Cotton Mills Company [8]. As a part of this test, bale quantities of cotton had been subjected to carefully controlled heating and cleaning conditions during ginning. The design of the 1957 California Gin Test and the evaluation of fiber properties, processing performance, and end-product quality have been reported and published in the literature [4].

The TBA value for each of the twelve cottons comprising the 1957 California Gin Test was determined on the samples as received (TBA_0) and after they had been subjected for 1 min. to a temperature of 200° C. in a laboratory oven (TBA_1). The quantities of interest are $\Delta TBA = TBA_0 - TBA_1$ and the ratio TBA_1/TBA_0. The 1-min. heat-

[1] Editor's note: The TBA value as defined here stands in an inverse relationship to the "TBA index" of Pan et al. [7] and to the "TBA value" used by Harrison and Marsh, p. 1070 this issue, which are both based on absorption rather than transmission.

TABLE I. TBA Values for 1957 California Gin Test Cottons

Cotton pair	High heat					Low heat				
	TBA_0	TBA_1	ΔTBA	TBA_1/TBA_0	Moisture at gin, %	TBA_0	TBA_1	ΔTBA	TBA_1/TBA_0	Moisture at gin, %
I	59.0	46.7	12.3	0.791	4.6	57.2	45.5	11.7	0.793	6.4
II	55.6	47.0	8.6	0.847	5.5	58.5	43.2	15.3	0.740	6.8
III	49.8	41.7	8.1	0.837	6.8	56.3	45.2	11.1	0.800	7.4
IV	57.7	46.2	11.5	0.802	2.9	59.4	44.4	15.0	0.748	5.6
V	55.3	45.5	9.8	0.821	1.7	55.6	44.6	11.0	0.802	5.1
VI	50.0	43.2	6.8	0.864	2.7	56.1	43.4	12.7	0.772	4.4
Pooled 95% conf. limit	2.7	2.6				1.7	2.3			
Average	54.6	45.0	9.5	0.827	4.03	57.2	44.4	12.8	0.776	5.95

ing time was chosen after consideration of Figure 1 and the heating time involved in the drying step during commercial ginning. While it is not possible to know the gin heating time exactly, it appears reasonable to estimate that the lint is subjected to high temperatures for no longer than 60 sec. The 1-min. laboratory heating increment places this procedure well in the sensitive region of Figure 1.

The data for the twelve cottons shown in Table I are tabulated in such a way that comparison may be made between paired cotton lots which received the same extent of overhead and lint cleaning. The only difference between the paired cottons is the extent of heating or drying and, consequently, the moisture content during ginning, which is also shown in Table I.

Each of the twelve cottons shows a decrease in TBA value as a result of a 1-min. laboratory heating at 200° C. The decrease in the TBA value (ΔTBA) and the ratio of the TBA values (TBA_1/TBA_0) are shown in Table I. It would be expected that the TBA_1/TBA_0 ratio would be closer to unity for the cottons which were subjected to high heat during ginning than for those which were subjected to milder heating during ginning. This is indeed the case in five out of six cotton pairs; the two cottons in Pair I gave the same TBA_1/TBA_0 ratio.

Unfortunately, the differences in the TBA_1/TBA_0 ratios within each pair of cottons are not sufficiently large to allow use of this method as a reliable ab-

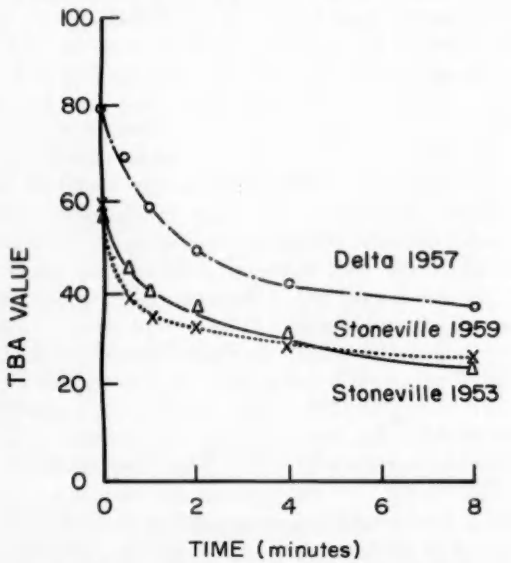

Fig. 1. Relation between TBA value and length of time heated at 200° C. for three cottons.

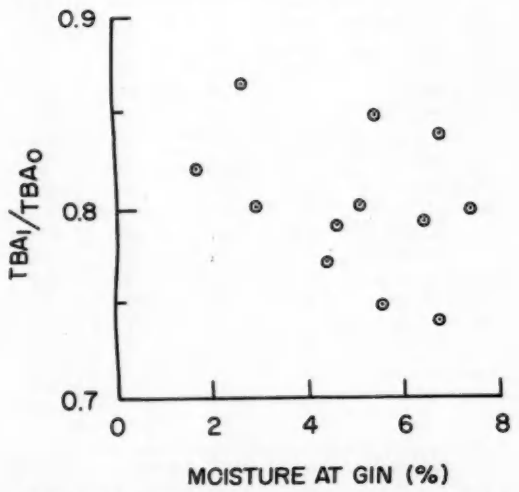

Fig. 2. Relative decrease in TBA value and the moisture content at the gin for 12 cottons from 1957 California gin test.

TABLE II. General Properties of Twenty Experimental Cottons

Cotton		Arealometer			Maturity, array method, %	Alkali scour weight loss, %
No.	Designation	A, mm.²/mm.²	D, mm.²/mm.²	Perimeter, μ		
20	Garo Hill	—	—	—	96	0.85
39	NR-AHA-C	324	8	48.4	98	1.35
13	Bobshaw IA	383	16	47.7	90	1.55
11	Cstlnd RNET	409	15	44.0	86	1.44
12	DPL 15	441	26	47.7	82	1.42
5	HA 46-124	434	15	41.4	90	2.18
21	CR4	402	19	47.7	85	1.79
55	Lengupa	352	14	50.0	92	2.31
38	Amsak	468	15	38.4	90	1.37
37	Pima S-1	458	15	39.2	90	1.36
54	De Ridder Red	326	12	52.2	94	1.68
3	THEF 96	510	25	40.7	82	3.18
53	Tanguis	396	14	44.4	90	1.86
52	Orig. Pima	509	32	44.4	80	2.17
35	Lankart	416	26	50.6	82	2.79
6	Acl. 442 (Br)	465	22	43.0	80	2.37
45	Acl. 1517C	459	26	46.0	84	2.65
49	SLS 1000	436	25	47.7	84	2.80
41	Acl. 442 (Sh)	476	32	47.4	78	3.04
36	Paymaster	465	32	48.5	75	4.26

TABLE III. TBA Values of Twenty Experimental Cottons

Cotton		TBA Value			
No.	Designation	Raw stock	Chloroform extracted	Ethanol extracted	Alkali scoured
20	Garo Hill	82.2	79.3	81.9	90.8
39	NR-AHA-C	79.6	83.1	83.6	87.6
13	Bobshaw IA	73.3	72.4	73.3	84.7
11	Cstlnd RNET	71.5	74.3	79.7	75.9
12	DPL 15	70.0	71.0	72.9	82.9
5	HA 46-124	68.4	69.3	77.8	74.9
21	CR4	68.4	66.6	71.2	91.0
55	Lengupa	68.4	67.8	70.8	84.8
38	Amsak	66.9	68.5	73.4	84.3
37	Pima S-1	66.4	67.2	72.6	81.6
54	De Ridder Red	65.8	65.3	73.6	87.7
3	THEF 96	63.1	61.0	75.8	72.5
53	Tanguis	59.0	60.1	62.6	83.8
52	Orig. Pima	57.3	62.9	67.1	80.5
35	Lankart	56.8	55.7	60.6	81.9
6	Acl. 442(Br)	56.5	57.3	70.0	78.7
45	Acl. 1517C	52.5	53.2	60.8	81.2
49	SLS 1000	47.0	50.1	62.6	82.3
41	Acl. 442 (Sh)	44.9	46.2	55.8	84.9
36	Paymaster	39.8	39.3	45.3	81.1
Pooled 95% conf. limits		2.7	2.1	2.8	2.2

solute diagnostic test for heat damage. Although in five out of the six cotton pairs the TBA_1/TBA_0 ratio is lower for the high heat cottons, there is no significant relationship between the TBA_1/TBA_0 ratio and the moisture content at the gin for the twelve cottons, as illustrated in Figure 2.

Differences among Cottons

The differences in the TBA_0 values among the three cottons shown in Figure 1 are real and experimentally reproducible. These differences cannot be ascribed to the effects of heat since the fibers were obtained on the seed and were hand-ginned in the laboratory. The differences may be due to the noncellulosic component of the fiber, to weathering in the field, or possibly to some structural characteristic of the cotton cellulose. It would be of interest to ascertain the cause of the differences among different cottons and to determine the suitability of the TBA value for characterizing cottons.

To investigate the differences in TBA values among cottons, twenty cottons were chosen from available samples on the basis of their differences in the fiber characteristics listed in Table II. The TBA values for these twenty cottons were measured in the following four conditions: (1) untreated, (2) chloroform-extracted, (3) ethanol-extracted, (4) alkali-scoured. The chloroform and ethanol extractions were performed in a Soxhlet apparatus, allowing at least 12 passes of the solvent. The alkali scouring was performed at 70° C. for 1 hr., using a solution containing 2% Na_2CO_3 and 2% NaOH.

The loss in weight during the alkali scouring treatment was determined in triplicate for each cotton and is also shown in Table II.

Examination of Table III reveals very large differences in the TBA value among the twenty cottons in the raw stock condition. Chloroform extraction does not significantly alter the TBA value, but ethanol extraction causes a slight increase. The increase in the TBA value as a result of ethanol extraction is particularly evident in those cottons whose raw stock TBA value is low. Alkaline scouring causes a large increase in the TBA value for all the cottons examined. In the case of alkaline scouring the increase in TBA value is again dependent upon the TBA value of the raw stock cotton. This is clearly evident in Figure 3 where the ratio of TBA value after alkaline scouring to the TBA value of cotton in the raw stock condition is plotted against the TBA value of the raw stock cotton. In view of the significant effect of the scouring treatment, the original TBA value was examined in terms of the weight losses caused by alkali scouring.

The relationship between loss in weight upon alakline scouring and the initial TBA value is shown in Figure 4. This highly significant relationship indicates that the interaction between 2-thiobarbituric acid and cotton is due to the noncellulosic component of the fiber which is removable by alkaline scouring and which is sensitive to thermal degradation.

In order to demonstrate graphically the effects of chloroform and ethanol extractions and of alkaline scouring, the twenty experimental cottons were separated into four groups, each containing cottons with similar TBA values in the untreated condition. Figure 5 shows how the average TBA values for

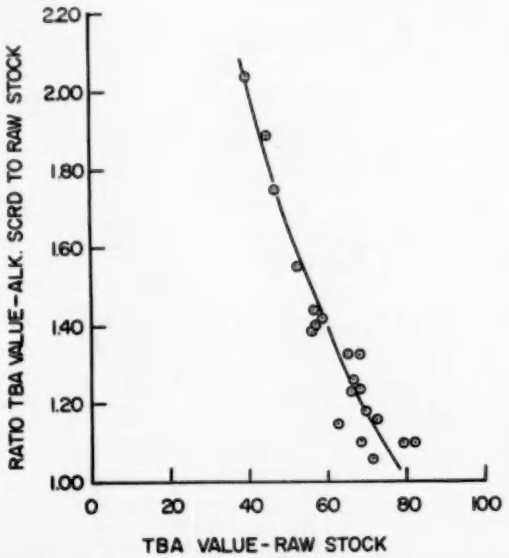

Fig. 3. Ratio of TBA value of alkali scoured cotton to TBA value of raw stock cotton vs. TBA value of raw stock.

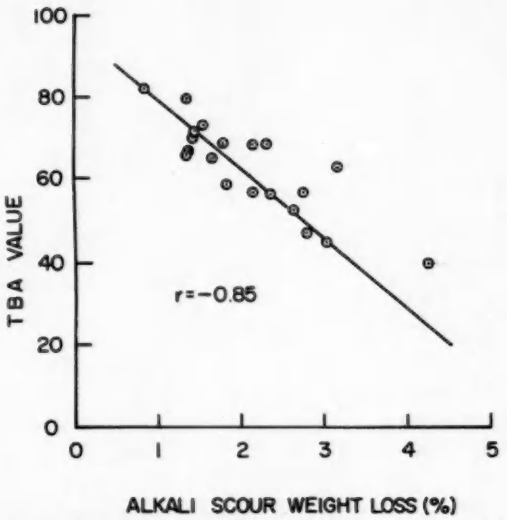

Fig. 4. Relation between TBA value and loss in weight upon alkali scouring.

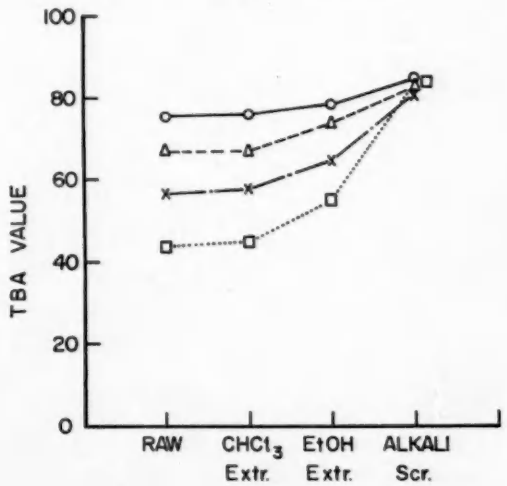

Fig. 5. Changes in TBA value as a function of cotton purification.

the four cotton groups change as a result of purification. The average loss in weight upon alkaline scouring for the four cotton groups is again a function of the original TBA value, as indicated in Figure 6.

It was of interest to examine whether any other fiber property is correlated with the TBA value. In Figure 7 is shown the statistically significant relationship between the TBA value and fiber maturity as determined by the array method. In view of the previously indicated relation between alkaline scour weight loss and the TBA value (Figure 4), the relationship shown in Figure 8 between alkaline scour weight loss and fiber maturity is to be expected. This relationship indicates that the more mature cottons have a lower noncellulosic component as measured by alkali scourable matter. For complete verification of this conclusion one must consider the total surface area of the different cottons and consider the possibility that the alkali scourable matter might be constant when expressed per unit surface area. As a measure of surface area one may consider the fiber perimeter as determined by the Arealometer [5]. When one expresses the alkali scourable matter per unit perimeter, the relationship with fiber maturity is statistically significant, as shown in Figure 9. This, then, clearly indicates that the more mature cottons have a lower noncellulosic component as estimated by alkali scourable matter. The possible use of the TBA value as a rapid measure of either fiber maturity or alkali scourable matter is evident from Figures 4, 6, and 7.

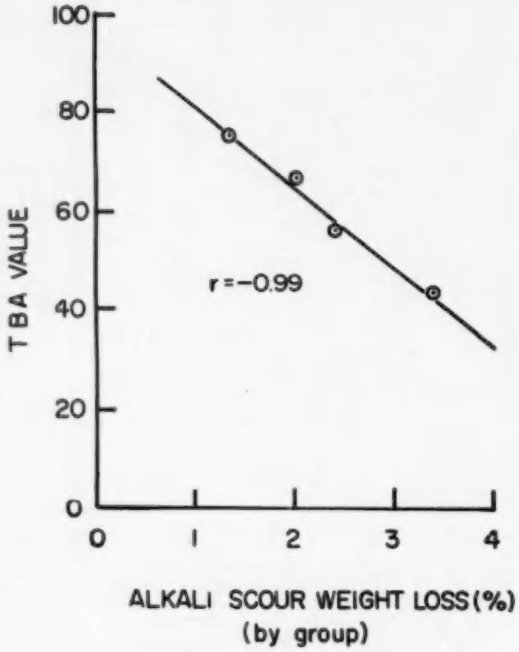

Fig. 6. Relation between TBA value and loss in weight upon alkali scouring (by groups).

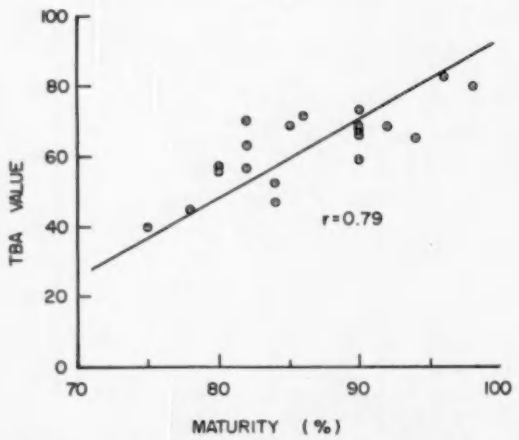

Fig. 7. Relation between TBA value and cotton fiber maturity.

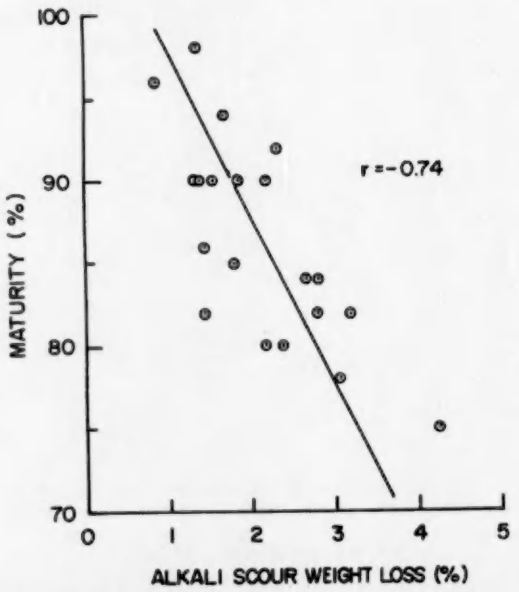

Fig. 8. Relation between cotton fiber maturity and loss in weight upon alkali scouring.

Conclusion

The relationships shown in this paper indicate that the TBA value may be used as a sensitive index of some forms of heat damage, as an index of alkali scourable matter, and as an index of fiber maturity. The major advantage of the test lies in the fact that it is extremely simple, rapid, and reproducible, and can be performed with a minimum of laboratory equipment. The major disadvantage of the test appears to be its lack of selectivity; many factors can contribute to changes in the TBA value. This disadvantage makes unambiguous interpretation of test results difficult. Despite this major disadvantage, the TBA value may prove to be of some use in the routine characterization of cotton. The data in this paper are presented with the intention of suggesting areas of possible applicability.

Acknowledgment

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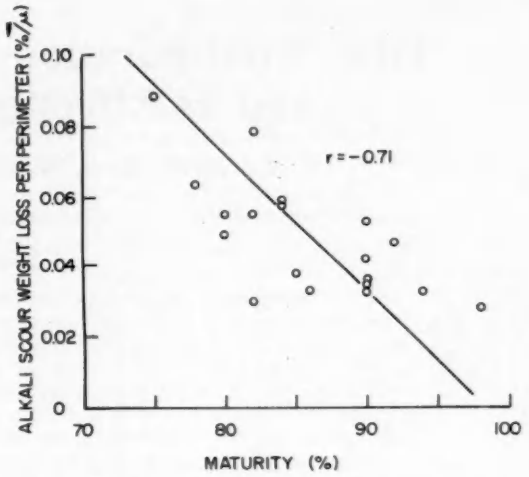

Fig. 9. Loss in weight upon alkali scouring per unit fiber perimeter vs. cotton fiber maturity.

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Fiber Configuration in Sliver and Roving and its Effect on Yarn Quality

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Abstract

Fiber configuration in sliver and the incidence of hooks in sliver and rovings have been studied, employing two different techniques. It has been established that during the drafting of sliver at draw frames, trailing hooks are removed preferentially as compared to leading hooks. The direction of presentation of hooks in the ingoing material (sliver or roving) at the ring frame has been shown to have a considerable effect on yarn strength and evenness. For best results with carded yarn, the majority of hooks in the feed to the ring frame should be presented to the drafting system in a trailing direction, i.e., there should be an odd number of processes between carding and spinning.

Introduction

Review of Literature

It is well known in the cotton spinning industry that fibers lie crisscross and entangled in a card sliver. In 1949, Morton and his associates [5] developed a tracer technique which enabled them to see the fiber as it lies in a sliver. This was a considerable advance over the indirect methods previously used by Löfgren [4] and Foster et al. [2]. Morton's data [5, 6] showed that (i) the majority of fibers in card sliver are hooked at either or both ends and that trailing hooks are more numerous than leading hooks and (ii) the process of drafting reduces the number and extent of hooks, until after the third passage of drawing a large majority of the fibers have no hooks. The third conclusion from the data, about leading hooks being preferentially removed during drafting, appears to be open to question.

Several workers have reported on the effects of parallelization in the material fed to a drafting system on drafting behavior or quality of output. The hypothesis that fiber parallelization helps better drafting was indirectly supported by the work of Löfgren [4] and Caveney and Foster [1]. Löfgren evolved an index of fiber parallelization based on the strength of slivers and determined the increase in irregularity of slivers with low and medium degrees of parallelization. His results showed that, other conditions remaining the same, the added irregularity is more at lower degrees of parallelization in the material fed. Caveney and Foster reported that the force required to draft a sliver is appreciably different in the two directions and that the difference can be attributed to the different fiber arrangement in the two directions. They found that the drafting force was less when the majority of hooks were fed as trailing. In these circumstances, the higher drafting force may be an indication of less controlled drafting and therefore of a more uneven output. The quality of output, therefore, may be expected to depend to an appreciable extent on the configuration of fibers in the ingoing material. Nutter [8] and Nutter and Slater [9] showed that the direction of feed of sliver or roving to the ring frame is of importance in determining yarn quality.

The present paper attempts to study more thoroughly the behavior of fiber hooks present in slivers and rovings during drafting and the effect of such hooks in the ingoing material at ring frames on yarn quality, particularly for the sliver-to-yarn spinning system. Section 1 of the paper deals with the configuration of fibers in slivers. The effect of fiber configuration on the quality of yarn spun on a sliver-to-yarn spinning frame is investigated in Section 2. Section 3 deals with fiber configuration in rovings, while Section 4 is devoted to the effect of this on yarn quality. Yarn quality has been expressed in terms of skein strength and yarn irregularity (Uster C.V.).

Methods of Determining Fiber Configuration

Two methods of determining fiber configuration have been used in the work reported here. By con-

figuration is meant the crimp or departure from straightness, type and extent of hooks, and the inclination of fibers to the sliver axis. The word "configuration" has been used in preference to "arrangement," used by Morton, because it is felt that the former is free from any suggestion of the "relative placement of fibres along the axis of a sliver." One of the methods of ascertaining configuration is a direct method of counting the number of hooked fibers, using a fluorescent-tracer-fiber technique. The second is an indirect method, first developed by Lindsley [3], which gives an index of fiber configuration called the "combing ratio." These methods have been fully discussed in a separate communication [10]. Brief descriptions of the methods used have been given at appropriate places in the paper.

Test Methods

All testing was done under standard atmospheric conditions of relative humidity, 65% ± 2%, and temperature, 80° ± 2° F.

Fiber length characteristics of cotton were determined from duplicate Baer Sorter patterns.

Fiber weight per unit length was determined in accordance with ASTM Standard Method D-1442-54.

Yarn skein strengths were determined in accordance with ASTM Standard Method D-1578-59T. A pendulum type of Goodbrand constant-rate-of-traverse (12 in./min.) machine was used.

For determination of skein strength, 30 skeins were tested in each case, 5 skeins from each of 6 bobbins. The pooled coefficient of variation of the mean for corrected skein tests was about 1.0%. A difference between two corrected mean skein strengths of about 2.8% can be considered to be statistically significant at the 95% confidence level. For purposes of comparison between different treatments, the skein strengths were corrected to nominal yarn count in accordance with ASTM Standard Method D-1578-59T.

The yarn irregularity was determined on the Uster Evenness Tester, Type B-17 with quadratic integrater ITGQ6. A material speed of 25 yd./min. was employed. For each treatment, eight bobbins were tested, two observations being taken from each bobbin. Each observation was the mean of five readings, taken at half-minute intervals. A difference in unevenness C.V. of about 0.75% can be considered to be significant at the 95% confidence level.

Section 1. Effect of Drafting on Fiber Configuration in Slivers

Experiment and Results

The following experiment was conducted to examine whether the direction of presentation of slivers to the drafting zone at drawing affects the extent of hook removal. Indian cotton C02 was used; its important fiber characteristics were: effective length, 1.10 in.; mean length, 0.90 in.; C.V. of fiber length, 32%; hair weight, 4.2 μg./in.

The cotton was carded on a standard MMC card of 40 in. width having flexible fillet on doffer and cylinder. The sliver number was 3950 tex (0.15's cotton count) and the rate of production was 10 lb./hr.

The card sliver was divided into two lots; each lot was drawn thrice on a Tweedles and Smalley four-over-four roller conventional drawing frame with a draft and doubling of six at every passage. The drafts in the three zones, back to front, were 1.24, 1.76, and 2.73. The sliver delivery speed was 90 ft./min.

One of the two lots of card sliver was processed in the orthodox way through three passages on the drawing frame. In such processing, the trailing and leading hooks in the sliver at any process are reversed and fed as leading and trailing to the next process. For example, the fibers which emerge from the card with hooks at the rear, that is, trailing hooks of card sliver, are fed to the first drawing frame with these hooks at the forward end with respect to the direction of motion of fibers at drawing. The other lot was reversed before being fed to the first drawing process so that the trailing and leading hooks of card sliver were fed again as trailing and leading hooks to the first drawing process. The first drawing frame sliver was then processed through two further passages of drawing in the orthodox way.

The number of each type of hook in the fibers constituting the card and drawing slivers was counted by the method indicated below.

Examination of hooks by fluorescent-tracer-fiber technique. A small proportion (0.25% by weight) of fibers dyed with a fluorescent dye was introduced into the blow room lap. Samples of card web were collected at random on blackboards. For draw frames, a ribbon corresponding to one sliver of the feed was taken from the delivered web on a blackboard. These boards were examined under an ultra-

TABLE I. Distribution of Hooked* Fibers in Card and Drawing Slivers

Sliver reference	Sliver from	Percentage† of fibers with: Leading hooks	Trailing hooks	Both ends hooked	No hooks (straight)	Others
	Card	19	47	8	24	2
	Normal feed to first drawing					
A	I Draw frame	41	6	4	47	2
B	II Draw frame	6	26	2	62	4
C	III Draw frame	23	4	1	70	2
	Reverse feed to first drawing					
D	I Draw frame	11	26	4	54	5
E	II Draw frame	26	8	2	62	2
F	III Draw frame	7	16	1	74	2

* Hooks are termed trailing or leading relative to the direction of delivery at that process.
† Percentages are rounded to the nearest integer.

violet lamp. The tracer fibers were counted and classified into groups similar to those used by Morton. For each material, six boards were collected at random. Each board contained about 90 tracer fibers so that the estimate of the percentage of hook types was in each case based on a classification of about 500 tracer fibers. The number of fibers having any particular type of hook can be considered to be a binomial variate. The statistical significance of the difference between two observed proportions can be tested, for the large samples taken, against twice the standard error of the difference, at a 95% confidence level [12]. The standard error of any observed proportion p is $\sqrt{pq/n}$ where $q = 1 - p$ and $n =$ numbe. of samples taken, about 500 in each case. The following differences between two observed val-luse of the percentages of hooks of any type can be considered to be significant:

Average level of percentage occurrence	Significant difference in percentage occurrence
15	4.5
30	5.8
50	6.3

The experimental results are given in Table I.

Discussion and Conclusions

The data from Table I, incidentally, confirm Morton's findings about card sliver. Most of the fibers in card sliver are hooked, the majority being hooked at the trailing end. The data also support his broad conclusion that the percentage of hooked fibers decreases and that of straight fibers increases with successive passages of drawing. But, as will be evident from the later discussion, the picture of hook removal as brought out by the present data is very different from that given by Morton. In determining whether the leading or the trailing hooks are preferentially removed during drafting, the fibers with both ends hooked have been added to each of them. This addition is based on the assumption that a trailing hook, say, will be removed equally readily whether or not that fiber has a hook at the other end. The reduction of hooks fed as trailing in a drafting zone is termed "trailer reduction" and the reduction of those fed as leading is termed "leader reduction." Hereafter the trailing and leading hooks of the parent card sliver are called major and minor hooks, respectively. It is known that trailing hooks of card sliver are larger in average extent than leading hooks. In orthodox processing, at the first drawing frame immediately after carding and at every alternate subsequent process, major hooks are fed as leading and minor hooks are fed as trailing, while at the second drawing frame and at every alternate subsequent process, major hooks are fed as trailing and minor hooks are fed as leading.

The data from Table I have been rearranged in Table II to facilitate analysis. It will be seen that the over-all trailer reduction in one passage of drawing is from 178, i.e., $(27 + 45 + 8 + 55 + 15 + 28)$, to 100, i.e., $(10 + 28 + 5 + 30 + 10 + 17)$, or, 44%. Similarly the over-all leader reduction is 20%. This evidence proves conclusively that the trailer reduction is much greater than the leader reduction.

The trailer and leader reductions computed sepa-

TABLE II. Removal of Hooks During Drafting

Machine	Percent hooks: Total leading	Total trailing
Card	*27	55 †
I Draw frame	† 45	10*
II Draw frame	* 8	28 †
III Draw frame	† 24	5*
Card	*27	55 †
I Draw frame	*15	30 †
II Draw frame	† 28	10*
III Draw frame	* 8	17 †

† Major hooks ⟶ Leader reduction
* Minor hooks ⟹ Trailer reduction

rately for major hooks turn out to be 33% and 14% while for minor hooks the reductions are 50% and 34%. It is evident that (i) trailer reduction is much greater than leader reduction for both major and minor hooks; (ii) minor hooks, whether fed as leading or trailing during drafting, are more readily removed than major hooks. Major hooks are especially difficult to remove if fed as leading during drafting.

A process can be called favorable to the removal of a hook if the latter passes through the drafting zone in a trailing direction. The extent of removal of either type of hook depends more on the number of passages favorable to the removal of that type of hook than it does on the total number of passages the material has been processed through. For example, though slivers C and F, in Table I, had three drawing passages each, the major hooks are fewer in F because it had an additional passage favorable for major hooks. The order in which favorable and unfavorable passages occur seems to be immaterial, as can be seen by comparing slivers B and E. Though the second passage was favorable to the removal of major hooks for B and the first passage for E, the total reduction in major hooks is the same for both. The same may be said about minor hooks also.

In order to confirm that the number of favorable passages determines the extent of hook removal, another experiment was conducted. Card sliver was suitably processed through three passages of drawing to obtain four types of sliver, in which the number of passages favorable to the removal of major hooks ranged from 0 to 3. About 500 tracer fibers were observed for each sliver. The data on hooks are given in Table III and require no comment.

At this stage of the discussion, it is necessary to point out that conclusion (i) above is apparently a complete contradiction of the conclusion drawn by Morton [6] from his data. Conclusion (ii), however, is the same as Morton's. It is interesting to note that Morton considers only the first-passage data and neglects the contribution of fibers hooked at both ends when he concludes that leader reduction is greater than the trailer reduction. Nield [7] has rightly taken account of both factors in analyzing the same data. A portion of that data, relating to the reduction of major hooks, is reproduced here.

TABLE III. Removal of Major Hooks vs. Number of Favorable Passages

Number of passages favorable to removal of major hooks	Percent hooks in 3rd passage sliver	
	Major	Minor
0	39	2
1	27	6
2	21	7
3	11	11

Nield's Interpretation of Morton's Data

Hooks as presented, %	Hooks remaining after drafting: Single passage, %	Double passage, %	Mean, %	Proportion of hooks removed, %
62 leading	30	37	33.5	46.0
62 trailing	41	23	32.0	48.5

According to Nield, "the fourth column shows the average effect of first and second passage" on hook removal. From the 5th column he concludes that there is no difference between leader and trailer reduction. The physical significance of this process of averaging is not clear. For instance, if x hooks are fed to the first passage which reduces them to y and, further, if these y hooks are fed to another passage which reduces them to z, then the "average effect" of a single passage of drawing will be obtained by comparing $x + y$ with $y + z$. It is fallacious to compare x with $(y + z)/2$ as has been done by Nield. When correctly analyzed, the proportions of hooks removed turn out to be 27% and 38% in the leading and trailing directions of presentation. Thus, Morton's data, when interpreted as above, do support the conclusions drawn from Table I.

Mechanism of Hook Removal

It seems reasonable to assume that a hook can be removed in a drafting zone only through frictional contact with other fibers moving at speeds different from that of the hooked fiber. The hook removal will be greater (i) the larger the number of such contacts at any instant and (ii) the greater the entanglement and cohesion with neighboring fibers moving at different speeds. It will be assumed, following Morton [6], that a hook will be removed only if the other end of the fiber is positively gripped by a nip and that floating fibers do not take any part in removing hooks. Further, let the drafting be ideal so that a fiber released from the back roller continues

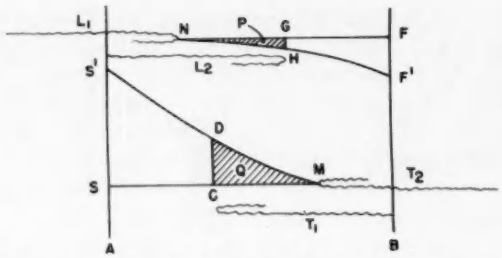


Fig. 1. Two positions each of a leading and a trailing hook identical in fiber and hook extent.

to move at that speed until its front end is gripped by the front nip. Figure 1 shows two positions each of a leading and a trailing hook identical in fiber and hook extent. A and B are the back and front roller nips, respectively. The figure also shows the slow and fast moving fiber beards $SS'M$ and $FF'N$ projecting into the drafting zone at any instant from the back and front nips respectively. The beard outlines are identical with the cumulative frequency curves of fibers of different lengths projecting from the nips. The bases of the two beards, SS' and FF' represent the number of fibers held by back and front nips, respectively. Hence $SS' = d \cdot FF'$, d being the draft. SM and FN represent the longest projecting fibers, $SM = FN$. Any section through the beards, parallel to the nips, represents the number of slow or fast moving fibers at that section. In actual fact, of course, the fibers are not arranged according to increasing length. The fibers in each beard are also being constantly replaced by fresh fibers.

Now consider a trailing fiber in the drafting zone. T_1 represents the position at which the fiber tip is gripped by the front roller. Up to this position the fiber was moving at the speed of the back beard and there was little possibility of its hook being removed. From position T_1 to position T_2 the fiber, moving fast, brushes against the slow moving fibers of the back beard and in this region the trailing hook can be removed. The number of slow moving fibers with which it is in contact diminishes steadily as it moves forward, the shaded area Q being proportional to the total number of contacts with slow moving fibers in the entire period during which the hook may be removed.

The area P has been similarly obtained. Unhooking possibility starts when the hook reaches N and continues till the section GH is reached when the fiber loses contact with the back nip. It can be seen that $MC = NG$ and $GH = DC/d$. Thus Area P = Area Q/d. The total number of effective contacts are thus apparently less for leading hooks than for trailing hooks. But since the speed of the fiber L_1 or L_2 is $1/d$ times the speed of the fiber T_1 or T_2, the total time during which effective contacts can take place on a leading hook is d times longer than that for a trailing hook. Morton [6] concludes therefore that, on this score, the probability of removal of a leading or a trailing hook is the same. It is suggested that this is not so. Probably more important than the total number of effective contacts, or the duration of the period over which a given hook is subject to contacts from other fibers, is the density of the effective fiber fringe which brushes past a hook or through which the hook brushes. This is more at any instant for the trailing hook, which therefore is removed more easily.

Morton further states that leading hooks are more easily removed because leading hooks are acted on by fast moving fibers in the region of the drafting zone (i.e., nearer the back nip) where fiber density, interfiber friction, and fiber entanglements are higher. Though the statement is correct, one must remember that most of the fibers in this region are slow moving and cannot therefore exercise an effective hook-removing action on the slow moving leading hooks.

Section 2. Effect of Fiber Configuration in Slivers on Yarn Quality

In the first phase of the work, the combing ratio was used as an index of fiber configuration in slivers. The sliver is clamped between lines A and B (Figure 2), and protruding fibers are cut along B. The fibers are then held only at A, and fibers not held at A are combed out in the direction AB. The protruding fibers to the left of A are cut away and discarded.

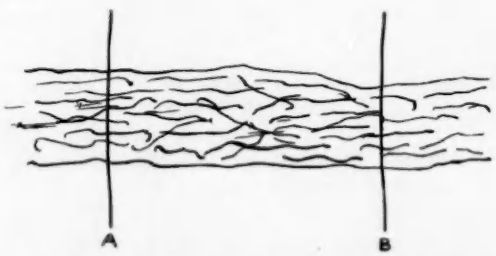


Fig. 2. Sketch showing clamp positions for determining combing ratio.

The weight of the combed portion divided by the weight of fibers held at A gives the combing ratio. If the sliver is held at B and combed in the direction BA, a combing ratio corresponding to the other direction of combing is obtained. The distance between A and B was $\frac{1}{2}$ in. for all measurements reported in this paper. As reported elsewhere [10], the combing ratio (hereafter denoted by CR) is a combined measure of fiber crimp, lack of parallelization, the number of hooks, and their extents. For a given sliver, the first two factors contribute equally to the CR's in two directions. But, as in card sliver, if the number of trailing hooks (and their extent) is greater than the number of leading hooks (and their extent), then the CR is greater for the forward direction (i.e., combing being done in the direction of delivery at cards) than for the backward direction. Henceforth, the forward and backward combing ratios will be referred to as CR_F and CR_B.

Experiment and Results

Card sliver of number 3030 tex (0.195's cotton count) from C02 cotton processed on the MMC card at 7.5 lb./hr. was passed in the orthodox manner through three passages of the Tweedles and Smalley drawing frame already referred to, using a draft and a doubling of 8 at every passage. The sliver delivery speed was 90 ft./min. The drafts in the three zones, back to front, were 1.24, 1.76, and 3.67. The drawing frame sliver from each of the passages was spun to 19.7 tex yarn (30's cotton count) on a Japanese OMS sliver-to-yarn spinning frame. The sliver was fed to the ring frame both in the orthodox way and after a reversal. The ring frame had a ring diameter of 2 in. and spindles of 10-in. lift; the spindle speed employed was 8000 r.p.m. The data on combing ratios of slivers and on yarn quality are summarized in Tables IV and V.

During preliminary investigations it was found that the CR of slivers increases when the slivers are stored. Therefore, the ratios were determined on the same day the slivers were produced, and the spinnings were carried out within two days of sliver preparation.

TABLE IV. Combing Ratios of Slivers

Sliver from	Combing ratio	
	* Forward	* Backward
Card	1.53	1.35
I Draw frame	0.82	1.04
II Draw frame	0.76	0.71
III Draw frame	0.65	0.71

* With respect to the direction of delivery at that process.

TABLE V. Yarn Quality

(Nominal number, 19.7 tex (30's); T.P.I., 25.0; ring frame draft, 160)

Yarn reference	Feed to ring frame sliver from	Direction of feed	Yarn quality	
			Corrected skein strength, lb.	Unevenness, Uster C.V., %
J	I Draw frame	Normal	60.0	24.9
K	I Draw frame	Reverse	47.7	27.5
L	II Draw frame	Normal	56.5	23.9
M	II Draw frame	Reverse	59.9	22.3
N	III Draw frame	Normal	62.5	22.2
O	III Draw frame	Reverse	58.3	22.6

TABLE VI. Hooked Fibers in Slivers and Yarn Quality

(Nominal number, 19.7 tex (30's); T.P.I., 25.0; ring frame draft, 205)

Sliver reference	Direction of feed	Corrected skein strength, lb.	Unevenness, Uster C.V., %	Percent hooks in feed	
				Leading	Trailing
A	Normal	57.3	23.7	6	41
	Reverse	50.2	26.5	41	6
C	Normal	62.0	21.9	4	23
	Reverse	58.7	22.0	23	4
D	Normal	55.7	23.8	26	11
	Reverse	59.1	22.7	11	26
F	Normal	61.0	22.8	16	7
	Reverse	63.6	20.9	7	16

In the second phase of the experiments, the percentage distribution of hooked fibers was observed directly. The slivers A, C, D, and F, of Table I, Section 1, were fed in normal and reverse directions to the same ring frame working at the same speed, and again yarn of 19.7 tex (30's) was spun in each case. The results of tests on yarn quality are given in Table VI. The last two columns of Table VI refer to the percentage of hooks fed as leading and trailing to the ring frame and have been taken from Table I.

Discussion and Conclusions

The data on combing ratios are sufficient to draw independent conclusions regarding preferential trailing hook removal during drafting, similar to those presented in Section 1. As seen from the analysis given overleaf, the percent reduction in CR corresponding to the hooks fed as trailing in a passage was more than that obtained for hooks fed as leading. Since CR is not a measure of hooks alone, the magnitudes of these reductions cannot be compared with those obtained using the direct method of counting

hooks, nor is it possible to analyze separately the effect on major and minor hooks.

	Percent reduction in CR		
Draw frame passage:	I	II	III
Hooks trailing	39.3	26.9	8.4
Hooks leading	32.0	13.4	6.6

The results of Table V are interesting as they demonstrate the importance of the degree of fiber parallelization and the percentage of hooked fibers in determining yarn quality. These will be considered below in detail.

First-passage sliver. The combing ratios are different for the two directions and hence the number and extent of hooks in one direction are different from those in the other direction. Correspondingly, the quality of yarn spun in one direction is expected to be better. In fact, the skein strength of J is 26% greater and its unevenness C.V. 2.6% less than that of K. In the first-passage sliver, the leading hooks were in majority ($CR_B > CR_F$) and for yarn J these were fed as trailing to the ring frame. The remarkable superiority of yarn J over yarn K shows that the majority of hooks should be fed as trailing at ring frame to obtain better quality yarn.

Second-passage sliver. The CR's are smaller than those for the first head sliver and in particular the difference $CR_F - CR_B$ is also much less. As a result, the yarn M is only 6% stronger than yarn L and the difference in Uster unevenness C.V. is 1.6%. When the sliver is reversed, the majority of hooks are fed as trailing and yarn M is better than yarn L.

Third-passage sliver. The CR's are still further reduced, the reduction being less than that caused by either the first or the second passage of drawing. The difference $CR_F - CR_B$ is about the same as that for second-passage sliver. As expected the difference in strengths of yarn spun in two directions is about the same (6.5%) as that obtained for second-passage sliver (6.0%). The difference of 0.4% in the Uster C.V. of yarns N and O is statistically not significant and hence should be taken as only indicative.

The close correspondence between the CR's of the sliver and the quality of yarn spun from them becomes evident when the data are rearranged as in Table VII.

The conclusion, based on the evidence of combing ratios, that the quality of yarn is better when the majority of hooks fed to ring frame are trailing is brought out distinctly by the data on hooks (Table VI). Each of the four slivers A, C, D, and F gave better yarn quality when the number of hooks fed as leading was smaller than the number of hooks fed as trailing.

When comparisons are made among slivers, the statement, "the smaller the number of hooks fed as leading, the better the quality of yarn," appears to hold for major hooks. The data from Table VI are rearranged in Table VIII. The percent minor hooks fed as leading and yarn quality do not seem to be correlated. This lack of correspondence may be attributed to one or more of the following factors: (1) below a certain percentage of hooks (say about 10%), the yarn quality may depend more on other factors than on the number of leading hooks fed to ring frame; (2) minor hooks, because of their smaller extents, may have little effect on yarn quality.

It may be noted that slivers processed through different numbers of passages and therefore having different degrees of parallelization were used in this comparison. In order to eliminate the effect of different numbers of drawing passages, data were obtained by spinning the four types of third-passage sliver (Table III) in normal and reverse directions. The results on yarn quality are presented in Table IX. It is of interest to note that here, too,

TABLE VII. Combing Ratio and Yarn Quality

	Average			Difference		
Drawing passage	CR of sliver	Corrected skein strength, lb.	Uster C.V., %	$CR_F - CR_B$	Skein strength, N*—R*, lb.	Uster C.V., %, N*—R*
I	0.93	53.8	26.2	0.22	12.3	2.6
II	0.73	58.2	23.1	0.05	3.4	1.6
III	0.68	60.4	22.4	0.06	4.2	0.4

* N = Normal feed; R = reverse feed.

TABLE VIII. Relation of Yarn Quality to Number of Hooks Fed as Leading

Major hooks			Minor hooks		
Percent hooks fed as leading	Corrected skein strength, lb.	Uster C.V., %	Percent hooks fed as leading	Corrected skein strength, lb.	Uster C.V., %
41	50.2	26.5	11	59.1	22.7
26	55.7	23.8	7	63.6	20.9
23	58.7	22.0	6	57.3	23.7
16	61.0	22.8	4	62.0	21.9

TABLE IX. Relation of Yarn Quality to Number of Hooks Fed as Leading

Major hooks			Minor hooks		
Percent hooks fed as leading	Corrected skein strength, lb.	Uster C.V., %	Percent hooks fed as leading	Corrected skein strength, lb.	Uster C.V., %
39	52.9	24.1	2	64.0	20.5
27	55.1	23.2	6	61.3	22.1
21	58.3	20.3	7	63.7	19.3
11	61.3	20.8	11	65.4	19.3

the correspondence between hooks fed as leading and yarn quality is good for major hooks but not for minor hooks.

One important feature of the data presented in Table V is the interpassage comparison of yarn quality. For yarns J, M, and N, major hooks were fed as trailing at the ring frame. The strengths of the yarns were 60.0, 59.9, and 62.5 lb. The strengths of yarns K, L, and O for which minor hooks were fed as trailing to ring frame were 47.7, 56.5, 58.3 lb. For the former (J-M-N) the use of the second passage of drawing causes no increase in strength while for the latter the improvement is remarkable. On the other hand, in the yarn from the third-passage drawing the improvement is slightly more for the former (J-M-N). This apparently inconsistent behavior can now be explained on the basis of the pattern of hook removal brought out in Section 1 and the conclusions of the preceding paragraph. As an illustration, the pair J-M may be compared with pair K-L. For J-M, the minor hooks were fed as leading at the ring frame. The sliver for M had one more passage of drawing where the direction of presentation of hooks was favorable to the removal of major hooks. Thus yarns J and M correspond to slivers which had about the same number of leading hooks and therefore had comparable strengths in spite of the difference in trailing hooks. On the other hand, for yarns K and L, the major hooks were fed as leading to the ring frame. These were reduced considerably in the second passage and therefore the number of leading hooks fed to ring frame was much smaller for L than for K; hence the superior quality of L. Similar arguments can be advanced for the effect of the third drawing passage.

It is interesting to note from the Uster C.V. % of yarns J-M-N and K-L-O that, in each case, the improvement effected by the second passage is more than that effected by the third passage. The extent of reduction in C.V. % brought about by each passage is greater for K-L-O than that for J-M-N.

From the results presented in this section it can be concluded that the quality of yarn obtained on the sliver-to-yarn spinning system depends on the direction of presentation of hooks in the feed. For any degree of parallelization of the feed sliver, as measured inversely by the mean combing ratio, the yarn quality is superior if major hooks are fed as trailing rather than as leading. If major hooks are fed as leading, the yarn quality decreases steadily with increase in the number of major hooks. For a given type of hook fed to the ring frame in a given way, i.e., trailing or leading, the yarn quality increases with parallelization of feed sliver. The increase in yarn regularity with increased parallelization at successive drawings is particularly noticeable with the second-passage drawing sliver.

The possible reason why superior yarn quality is obtained when major hooks, which are more numerous and larger in extent than minor hooks, are fed as trailing is that, as in drawing, the ring frame drafting removes and straightens trailing hooks more easily than leading hooks, so that there are more unhooked fibers in the yarn to contribute to yarn quality. Since under these circumstances there is less "doubling back" of fibers, there is greater uniformity in the number of fibers per cross section and hence an improvement in yarn regularity. The average fiber extent is also longer and so contributes to higher strength. When, on the other hand, major hooks are fed as leading, not only is the fiber extent less and doubling back of fibers more in the yarn, but the very presence of such hooks with decreased fiber extents may make the process of drafting less controlled.

Section 3. Fiber Configuration in Rovings

The main object of this phase of the investigation was to determine the fiber configuration in rovings in the broad sense of twisted fiber strands, about which limited data are available in the literature [11]. It is also of importance to know up to what

stage of processing or up to what total draft the effect of direction of feed persists.

Experiment and Results

Card sliver from C02 cotton was processed into rovings, using the different sequences shown in the accompanying Processing Chart. The number of each type of hook in the rovings as well as in second- and third-passage drawing-frame slivers was counted. The card sliver number was 3950 tex (0.15's cotton count) and the final roving number in each case was about 330 tex (1.80's cotton count). The processing particulars up to drawing were the same as for the slivers in Table I. Platts' conventional slubbing and intermediate frames with 3-over-3 roller graduated drafting systems were used. The twist multipliers in slubbing and intermediate rovings were 1.10 and 1.25, respectively, and the spindle speeds employed on these frames were 650 r.p.m. and 750 r.p.m. The high draft roving frame was of the Platts' MS2 type with Casablancas four-roller, two-zone drafting system, a pair of aprons being provided in the front zone. The drafts used in the back and front zones were 2.80 and 4.25, with a tension draft in the middle zone. The spindle speed was 920 r.p.m. and the twist multiplier in the roving was 1.25.

The technique of counting tracer fibers was slightly modified for the rovings. Short pieces of roving were gently untwisted and placed on blackboards. When a tracer fiber was located, the fibers around it were gently teased away till it was easy to ascertain the exact configuration of the tracer. The method of sampling was the same as for slivers. Results are given in Table X.

The data on hooks in draw frame slivers were collected only to get a general idea about the material from which rovings were prepared. About 100 tracer fibers were counted for each sliver and hence the percentages of hooks in slivers are approximate estimates. More than 500 tracers were counted for each roving. For each of the rovings Q, R, and S there was one process at the fly frames favorable to the removal of major hooks. These hooks were fed as trailing to the intermediate frame for Q, to the MS2 high draft roving frame for R, and to the slubbing frame for S. The percentage reduction in major hooks from draw frame sliver to the final roving is 45% for Q, 52% for R, and 32% for S, and is highest for R where the draft at the favorable process was maximum. In each case the reduction is quite substantial, whereas the number of minor hooks hardly shows any change during processing at fly frames for these three rovings.

For roving P the single high draft roving frame after drawing was not favorable to removal of major hooks which were fed as leading to the drafting zones. Curiously enough, even here the reduction in major hooks was quite substantial, being 45%.

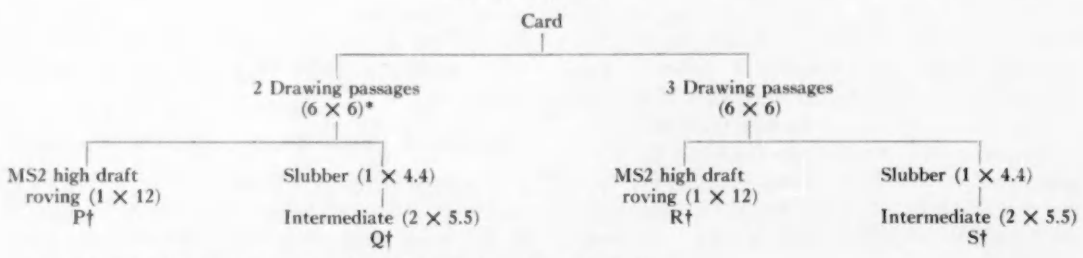
While no conclusions are being drawn from the limited data on the pattern of removal of leading and trailing hooks during drafting at fly frames, it is interesting to note that even for roving S, for which there were five processes of drafting after carding with a total draft of over 5000, there were still a substantial number of major hooks in the material fed to the ring frame.

Section 4. Effect of Fiber Configuration in Rovings on Yarn Quality

Experiment and Results

The four rovings P, Q, R, and S mentioned in Section 3 were each fed to a ring frame in two directions, in the orthodox way and after an artificial

Processing Chart


Card
2 Drawing passages (6 × 6)*
3 Drawing passages (6 × 6)
MS2 high draft roving (1 × 12) P†
Slubber (1 × 4.4)
Intermediate (2 × 5.5) Q†
MS2 high draft roving (1 × 12) R†
Slubber (1 × 4.4)
Intermediate (2 × 5.5) S†

* Figures in the parentheses refer to doublings and draft at that process.
† Roving reference.

reversal. Yarn of number 19.7 tex (30's cotton count) was spun in each case. The ring frame used was a Platts' frame equipped with an A-500 Casablanca drafting system. The spindle lift and ring diameter were 8 in. and 2 in., respectively, and the spindle speed was 9000 r.p.m. Results are given in Table XI.

Discussion and Conclusions

For a given roving, the quality of yarn was better when the majority of hooks were fed as trailing. For all the four spinnings where the major hooks were fed as trailing to the ring frame, the yarn quality was about the same. The yarn strengths varied between 58.9 lb. and 60.3 lb. and the yarn unevenness between 21.3% and 22.9%, only the latter figure being slightly higher than the other three. When the major hooks were fed as leading, the yarn quality, for spinnings P (reverse), R (normal), and S (reverse) was substantially lower than when the major hooks were fed as trailing. The maximum strength of yarn in these three spinnings did not exceed 55.5 lb., and the minimum uneveness was as high as 24.5%. However, for this type of feed, spinning Q (normal) proved to be an exception, with a yarn strength as high as 59.0 lb., though the yarn evenness (Uster C.V. 23.1%) was not good.

Since the number of major hooks was not very different for the four rovings and because of the exceptional behavior of yarn Q (normal) it is not possible to draw conclusions regarding the correspondence between yarn quality and the number of major hooks fed as leading to the ring frame. It may be noted, though, that if yarn Q (normal) is ignored, the maximum strength of yarn for major hooks fed as leading (55.5 lb.) was obtained when the number of major hooks was a minimum for spinning R (normal).

It may be concluded from the discussion that the direction of feed of roving to the ring frame has an important bearing on yarn quality. It is preferable to feed the major hooks as trailing. This can be achieved for a carded yarn if there is an odd number of processes between carding and spinning.

Summary of Conclusions

Card Sliver

1. Most fibers in a card sliver are hooked at one or both ends; of these, a large majority are hooked at the trailing end.

TABLE X. Percent Hooks in Slivers and Rovings

Sliver from	Percent fibers with *Leading hooks	*Trailing hooks	No hooks	Both ends hooked + others
II Draw frame	9	31	51	9
III Draw frame	25	5	65	5
Roving reference				
P	17	4	75	4
R	4	12	82	2
Q	7	17	74	2
S	17	7	75	1

* Relative to the direction of delivery at the process.

TABLE XI. Fiber Configuration in Rovings and Yarn Quality

Nominal number 19.7 tex (30's); t.p.i., 247; draft, 17.3

Roving reference	Direction of feed	Yarn quality: Corrected skein strength, lb.	Yarn quality: Unevenness, Uster C.V., %	Percent hooks as fed to ring frame: Leading	Percent hooks as fed to ring frame: Trailing
P	Normal	58.9	22.9	4	17
	Reverse	47.4	25.0	17	4
R	Normal	55.5	24.5	12	4
	Reverse	60.5	21.8	4	12
Q	Normal	59.0	23.1	17	7
	Reverse	60.3	22.1	7	17
S	Normal	60.3	21.3	7	17
	Reverse	52.9	25.0	17	7

Hook Removal in Draw Frames

2. With successive passages of drawing, the percentage of hooked fibers is reduced and that of fibers without hooks increased.

3. The trailer reduction at each passage is much greater than the leader reduction for either type of card hook.

4. The card leading hooks are more readily removed for any given direction of presentation than the card trailing hooks. The latter are especially difficult to remove if fed as leading during drafting.

5. The total reduction in hooks, leading or trailing in card sliver, depends more on the number of passages favorable to the removal of that type of hook than on the total number of passages. The order in which favorable and unfavorable passages occur is of little importance.

Fiber Configuration in Sliver and Yarn Quality in Sliver-to-Yarn Spinning System

6. For any given sliver feed, the yarn quality is better if card trailing hooks are fed as trailing to the ring frame than if they are fed as leading.

7. For any type of hook presented to the ring frame in a given way, the yarn quality increases with increase in fiber parallelization in the feed, the parallelization being measured inversely by the mean combing ratio.

8. If card trailing hooks are fed as leading to the ring frame, the yarn quality decreases with increase in the number of such hooks. No such correspondence is noted for card leading hooks.

Fiber Configuration in Rovings and Yarn Quality

9. The differential effect of the direction of feed at ring frame on yarn quality persists even after five preparatory processes subsequent to carding. It is preferable to feed the majority of hooks as trailing to the ring frames. This can be achieved by having an odd number of passages between card and ring frames.

Acknowledgments

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The Effect of pH and Detergents on the Felting of Shrink-Resistant Wool

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Abstract

The rate of felting of an untreated wool fabric increases with decreasing pH of the felting solution over the pH range 2–10. The felting rate is increased at all pH values by the addition of nonionic detergent to the solution.

The felting, relative to untreated wool, of wool made shrink-resistant with permanganate/salt, acid chlorine, permonosulfuric acid followed by sulfite, permanganate/hypochlorite, peracetic acid/hypochlorite, hypochlorite at pH 8, or gaseous chlorine increases with decreasing pH of the felting solution. The magnitude of this effect can be reduced by treatment with higher concentrations of reagents. After treatment with reagents of the concentrations used industrially, no change in felting rates with pH can be detected at pH values above 5. The actual rate of felting of wool treated with reagents of these concentrations is low and is not significantly affected by the addition of soap or nonionic detergents to the solutions.

The felting, relative to untreated wool, of wool treated with ethanolic thioglycolate, aqueous bisulfite (on extracted wool), hydrogen peroxide followed by bisulfite, ethanolic sodium hydroxide, or sodium hydroxide/saturated salt does not vary with pH in the absence of detergent. Felting is increased by the addition of detergent to the solutions. In detergent solutions, the relative felting of wool treated with ethanolic thioglycolate, aqueous bisulfite (on extracted wool), or hydrogen peroxide/bisulfite does not change with pH, but the relative felting of wool treated with sodium hydroxide in ethanol or saturated salt solution reaches a maximum at pH 7–9.

Frictional properties of fibers treated with some of the reagents have been measured (by the capstan method using a keratin rod) at different pH values in the presence and absence of detergent. Felting behavior does not appear to be explained by these frictional properties alone. Some other property, or properties, of treated wool, which vary differently with pH than do similar properties of untreated wool, must play a part in felting.

Introduction

Several curves relating the felting shrinkage of wool to the pH of the felting liquor have been published [3, 11, 12, 16, 20, 21]. There is some disagreement as to the shape of the curve in alkaline solutions, which may be due to the buffer solutions used [11, 20], the presence of small amounts of absorbed soap [11] (since soap and other lubricants on wool can significantly increase the rate of felting [6, 16]), to soap or detergents in the liquor, and probably also to differences in yarn structure, fabric structure, and differences in the forces applied in the various felting methods used. A higher felting rate in the presence of soap or detergents is probably to be expected on the basis of a lubrication effect. The frictional coefficients (anti-scale and with-scale) of wool are lower in sodium oleate than in liquor at the same pH without lubricant, although the difference between the frictional coefficients is the same [7].

It seems certain that felting is faster (at similar detergent concentrations) in acid than in neutral or alkaline solution [3, 11, 12, 16, 20, 21], the increased felting rate being paralleled both by a larger difference between frictional coefficients [13, 14, 20, 23] and by a reduced resistance to extension [19, 21]. Each effect could lead to an increased felting rate [20, 21].

The effect of pH of the liquor on the felting of shrink-resistant wool has also been investigated [1, 2, 12] and the results presented in two different ways. On comparison with a given area shrinkage

of an untreated sample after felting at different pH values, it has been found that wool treated with certain concentrations of acid chlorine, gaseous chlorine, chlorine in carbon tetrachloride, sulfuryl chloride, or ethanolic potassium hydroxide showed the same degree of shrink resistance at pH 3 and pH 10, but that wool treated with fluorine, acid permanganate, bromine in carbon tetrachloride, aqueous bromine, or alkaline hypochlorite felted at about the same rate as untreated wool at pH 3, although showing good shrink resistance at pH 10 [1]. The shrink resistance brought about by the latter group of reagents in acid solution was improved by treating with higher concentrations of the reagents [1]. These tests were carried out in the absence of detergent at pH 3 and in the presence of sodium oleate at pH 10, so the two sets of results are not strictly comparable.

In another study, the shrinkage–pH curves, for a given time of felting, of sulfuryl chloride-treated and ethanolic potassium hydroxide-treated wool were determined in buffer solutions in the absence of detergents [12]. The curve for ethanolic potassium hydroxide-treated wool was approximately parallel with that for untreated wool at pH 3 or above, but below pH 3 the relative felting of the treated wool was increased. The relative felting of the sulfuryl chloride-treated wool was lower in acid than in alkaline solution.

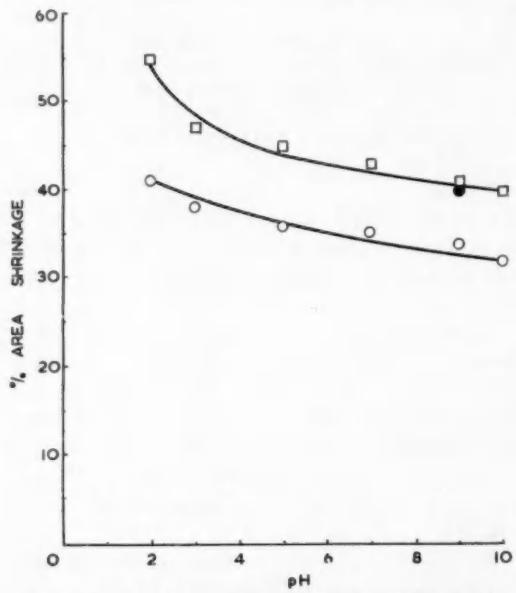

Fig. 1. Area shrinkage of untreated wool fabric after washing for 20 min. ○, no detergent; □, 0.05% (wt./vol.) Nonion C; ●, 0.05% (wt./vol.) soap solution.

The results appear to be qualitatively related to frictional properties of the treated wool. Acid permanganate-treated fibers have a low frictional difference (the difference between the anti-scale and with-scale frictional coefficients) at pH 10 but approximately the same frictional difference as untreated fibers at pH 3 [1]; acid chlorinated wool and sulfuryl chloride-treated fibers have a low frictional difference in both acid and alkaline solution [13, 14]. Changes in the frictional difference of fibers treated with ethanolic potassium hydroxide also roughly agree with changes in the felting behavior [14].

The felting of shrink-resistant wool at different pH values and in the presence and absence of detergents is of both technical and theoretical interest. The results in this paper extend previous work to a wider range of treated wools.

Experimental

Materials

Wool. A scoured, plain-weave worsted fabric (13 ends, 12 picks/cm., 2/30 tex yarn with both singles and two-fold twists of 3.9 turns/cm.) made from Merino 64's wool was used.

Reagents. All chemicals used were of ordinary laboratory reagent quality.

Methods

Shrink-resist treatments. These were generally carried out by treating 5-yd. samples of fabric in a small winch dyeing machine.

Felting determinations. Area felting shrinkages on 9-in. squares of fabric were determined after washing in buffer solutions at 45° in a machine, described previously as Machine E [10], running at full speed. The load in each test was made up to 1000 g. with 9-in. squares of cotton fabric. Before each washing test, the load and wool samples were allowed to stand in the appropriate buffer solution for at least 2 hr. The buffer solutions used were: pH 2.0, potassium chloride plus hydrochloric acid; pH 3.0, pH 5.0, and pH 7.0, disodium hydrogen phosphate plus citric acid; pH 9.0, potassium dihydrogen phosphate plus sodium borate; pH 10.0, boric acid plus sodium hydroxide.

Tests were carried out in the buffers in the pres-

ence and absence of 0.05% (wt./vol.) Nonion C (a nonionic detergent supplied by Gardinol Chemical Co.) and a separate test was carried out for each treated sample with 0.05% (wt./vol.) soap solution (sodium stearate, pH 8.9–9.1) in the absence of buffer salts.

Frictional properties. These were measured on fibers withdrawn from the yarns of treated or untreated fabric. The capstan method of Lipson and Howard [8], using a keratin rod, was used. The fibers and rod were lubricated by the appropriate buffer solutions with or without 0.05% (wt./vol.) Nonion C.

Results

Felting of Untreated Wool

The shrinkage–time curves for untreated fabric were determined over the range pH 2–10 in the presence and absence of detergent. (The shape of the curves under all conditions was similar to that already described for this, *untreated*, fabric in this washing machine [10]—that is, there was an initial straight line followed by a falling off in rate with time.) The area shrinkages after washing for 20 min. are given in Figure 1; similar curves are obtained if area shrinkages after any other times are plotted against pH. These curves agree roughly with some others reported [3, 11, 12, 16, 20] and show the expected increased felting rate in acid solution.

Increased rates in the presence of detergent are significant. The initial rate of felting at pH 9 in the absence of detergent is 1.8% area shrinkage/min. compared with 2.2% area shrinkage/min. in 0.05% soap solution or 0.05% Nonion C in the pH 9 buffer solution. The difference due to detergent is more marked at pH 2, initial felting rates being 2.2% area shrinkage/min. and 2.8% area shrinkage/min. in the absence and presence of detergent respectively.

Felting of Wool made Shrink-Resistant by Industrial Oxidation Processes

The shrinkage–time curves of wool fabrics *treated for a reasonable degree of shrink resistance* differ from those of *untreated* wool [10]. They consist of an initial, almost flat portion followed by an increase in slope before the typical falling off in rate, as the maximum area felting shrinkage is approached, occurs. The felting behavior of the *treated* wool can

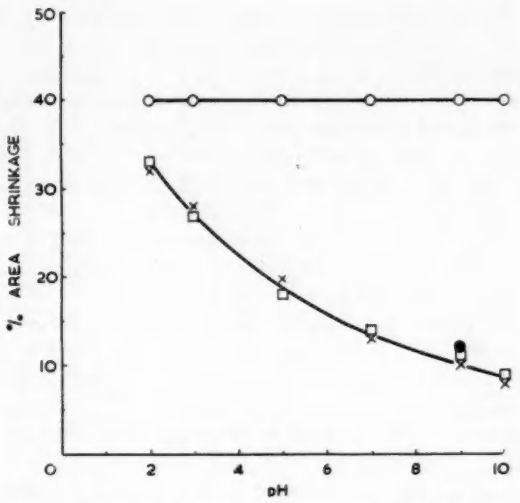

Fig. 2. Area shrinkages of wool treated with 3.5% permanganate/salt [9]. ○, untreated wool in presence and absence of detergent; □, treated wool in absence of detergent; ×, treated wool in 0.05% (wt./vol.) Nonion C solution; ●, treated wool in 0.05% (wt./vol.) soap solution.

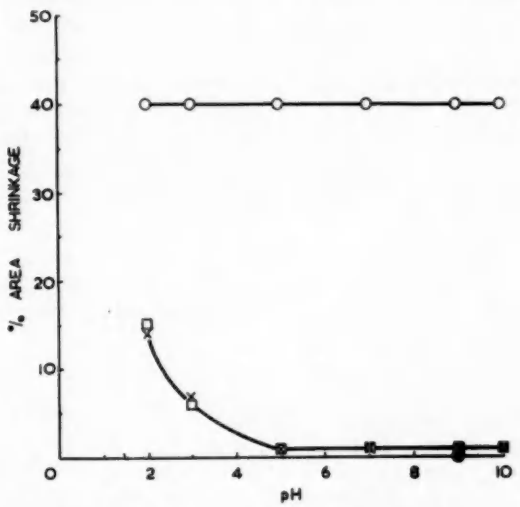

Fig. 3. Area shrinkage of wool treated with 5% permanganate/salt [9]. ○, untreated wool in presence and absence of detergent; □, treated wool in absence of detergent; ×, treated wool in 0.05% (wt./vol.) Nonion C solution; ●, treated wool in 0.05% (wt./vol.) soap solution.

be expressed in terms of slopes of the initial flat portions of the curves together with the slopes of the steeper second, approximately straight, stages of the curves [10], as area shrinkage for a given area shrinkage of untreated fabric, or as area shrinkages

after a given time of felting. The simplest way of presenting the results depends on the effects being investigated.

To find the effect of pH on the felting of treated wool, area shrinkage was plotted against pH for the various times at which untreated fabric had shrunk 10%, 20%, 30%, and 40% in area. These times depended on the pH of the felting liquor. All the curves were of similar shape for any one treatment, so only the results for times at which untreated had shrunk 40% are given.

The results for wool treated with 3.5% permanganate/salt [9] are given in Figure 2. There is a continual increased felting with decreasing pH of the felting liquor, an effect still observed, but to a lesser extent, when wool has been treated with 5% permanganate to give the degree of shrink resistance often required industrially. The curve for 5% permanganate/salt can be seen from Figure 3 to be approximately flat for washing liquors of pH 5–10.

Results with permanganate/hypochlorite [17], acid chlorine (applied by the bromate/salt [9] method), permonosulfuric acid [22], peracetic acid/hypochlorite [5], hypochlorite at pH 8 [4], and gaseous chlorine [24] are all similar to those found with permanganate/salt when wool has been treated to show about the same degree of shrink resistance. Concentrations of the various reagents which give equivalent shrink resistance have been given previously [10]. Figures 4, 5, 6, 7, 8, and 9 show typical curves for wool treated with the industrially used oxidizing agents. As with permanganate/salt, higher concentrations of all the reagents will considerably decrease the felting at the lower pH values.

Detergent in acid solutions was found to have about the same effect on the rate of felting of both untreated and treated wool. Therefore, as shown in Figures 2–9, detergent has had no effect on the relative shrinkages of untreated and treated fabrics. The felting rates are all the same in 0.05% soap and in 0.05% Nonion C in pH 9 buffer solution. For practical purposes, where wool has been made adequately shrink-resistant and laundering is above pH 5, the rates of felting of treated fabrics are low and are the same in the presence and absence of detergent.

Felting of Wool Treated with Other Shrink-Resist Processes

Farnworth [6] has reported that both treatment of wool with ethanolic thioglycolate and the reaction of aqueous bisulfite with ethanol-extracted wool give shrink resistance when the fabrics are tested in the absence of detergent. When detergents are present in the washing liquor, relative felting of the treated wool is increased. Treatment with hydrogen peroxide followed by a relatively severe bisulfite treatment has now been found to give a similar effect, some typical results being given in Table I. Treatment with sodium hydroxide in saturated salt solution also gives a shrink-resist effect when the treated

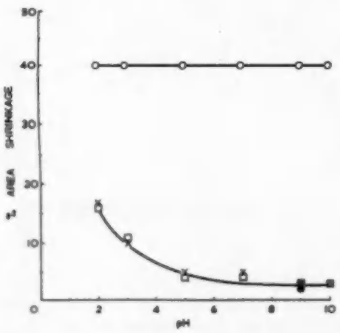

Fig. 4. Area shrinkage of wool treated with 2% permanganate + hypochlorite containg 2% active chlorine [17]. ○, untreated wool in presence and absence of detergent; □, treated wool in absence of detergent; ×, treated wool in 0.05% (wt./vol.) Nonion C solution; ●, treated wool in 0.05% (wt./vol.) soap solution.

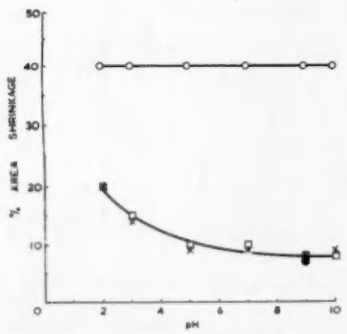

Fig. 5. Area shrinkage of wool treated with 1.8% acid chlorine by bromate/salt method [9]. ○, untreated wool in presence and absence of detergent; □, treated wool in absence of detergent; ×, treated wool in 0.05% (wt./vol.) Nonion C solution; ●, treated wool in 0.05% (wt./vol) soap solution.

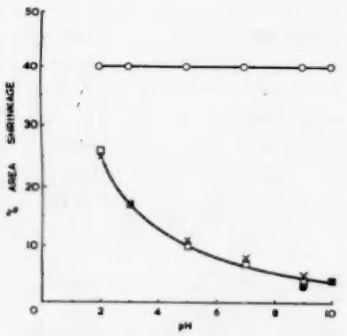

Fig. 6. Area shrinkages of wool treated with 1.8% permonosulfuric acid followed by sulfite [22]. ○, untreated wool in presence and absence of detergent; □, treated wool in absence of detergent; ×, treated wool in 0.05% (wt./vol.) Nonion C solution; ●, treated wool in 0.05% (wt./vol.) soap solution.

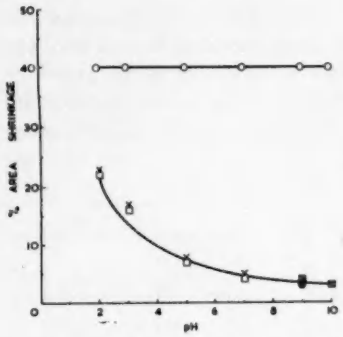

Fig. 7. Area shrinkages of wool treated with 1% peracetic acid and hypochlorite containing 2.5% active chlorine [5]. ○, untreated wool in presence and absence of detergent; □, treated wool in absence of detergent; ×, treated wool in 0.05% (wt./vol) Nonion C solution; ●, treated wool in 0.05% (wt./vol.) soap solution.

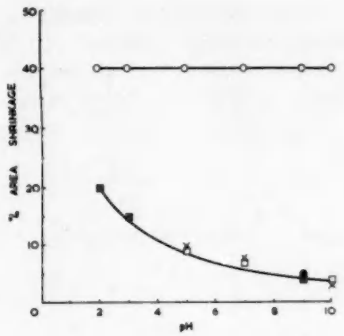

Fig. 8. Area shrinkages of wool treated with hypochlorite, containing 5% active chlorine, at pH 8 [4]. ○, untreated wool in presence and absence of detergent; □, treated wool in absence of detergent; ×, treated wool in 0.05% (wt./vol.) Nonion C solution; ●, treated wool in 0.05% (wt./vol.) soap solution.

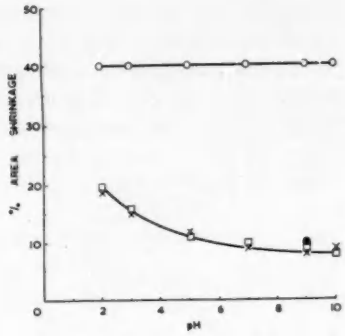

Fig. 9. Area shrinkages of wool treated with 1.0% gaseous chlorine [24]. ○, untreated wool in presence and absence of detergent; □, treated wool in absence of detergent; ×, treated wool in 0.05% (wt./vol.) Nonion C detergent; ●, treated wool in 0.05% (wt./vol.) soap solution.

wool is tested in the absence of detergent but, in soap solutions, the treated wool felts more rapidly than the untreated.

These treatments are therefore in a different category from the oxidations described in the previous section. In addition, it has been found that the felting of ethanolic sodium hydroxide-treated wool is significantly increased by addition of detergents to the washing liquor. The effect of pH on the extent of felting, for 40% area shrinkage of untreated samples, for these five treatments is shown in Figures 10, 11, 12, 13, and 14.

Fabrics treated with ethanolic thioglycolate, aqueous bisulfite on extracted wool, and peroxide/bisulfite show no variation in felting with pH, relative to untreated wool. These results are given in Figures 10, 11, and 12, and the effect of detergents is clearly shown.

Fabrics treated with sodium hydroxide in saturated salt or in ethanol also show no change in relative extent of felting with pH in the absence of detergents (Figures 13 and 14), but in the presence of detergents there is a maximum felting rate at pH 7–9. Sodium hydroxide/salt-treated wool felts more rapidly than untreated wool in detergent solutions in the pH range 6–10.

Within experimental error, felting rates for all treatments are the same in soap solution and in detergent solution at pH 9.

TABLE I. Shrink Resistance of Wool Treated with Hydrogen Peroxide Followed by Bisulfite

Peroxide treatment*	Bisulfite treatment	% Area shrinkage after washing in: pH 9 buffer	% Area shrinkage after washing in: 0.05% (wt./vol.) soap solution
(Untreated wool)		(40)	(40)
—	5% (wt./vol.), 50°, 2 hr.	38	42
5 vol., 80°, 2 hr.	—	24	28
2 vol., 50°, 2 hr.	1% (wt./vol.), 50°, 1 hr.	17	33
5 vol., 50°, 2 hr.	1% (wt./vol.), 25°, 1 hr.	14	32
5 vol., 50°, 2 hr.	1% (wt./vol.), 50°, 1 hr.	11	30
5 vol., 50°, 2 hr.	5% (wt./vol.), 50°, 2 hr.	9	29
5 vol., 80°, 2 hr.	1% (wt./vol.), 50°, 1 hr.	5	12

* All peroxide treatments were in 0.2% (wt./vol.) sodium pyrophosphate solution at pH 9.

Frictional Properties of Treated Wool

Results of friction measurements on untreated and some treated fibers are given in Table II. The frictional coefficients of untreated fibers are lower in the presence of detergent than when detergent is absent, but the frictional difference is the same in both cases. The frictional coefficients of untreated fibers in the presence of detergent are higher at pH 3 than at pH 9, and the frictional difference is also higher at the lower pH (significant at 1% level). These results agree with those of Makinson [15] for fibers which have not been solvent extracted.

The frictional properties for two examples of treated wool, where felting increases with decreasing pH and where the relative felting is unaffected by

detergent, are shown in Table II. In these cases (1.8% acid chlorine and 3.5% permanganate/salt) the frictional coefficients are lowered by the addition of detergent but the frictional difference, which is lower than that of untreated wool, is not affected. At pH 3 compared with pH 9, the frictional coefficients of 3.5% permanganate/salt-treated wool are higher, and the frictional difference is the same.

For treatments where felting is considerably affected by detergent (sodium hydroxide/salt and aque-

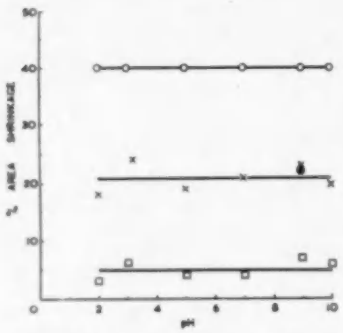

Fig. 10. Area shrinkages of wool treated with 1% thioglycolic acid + 6% ammonia in ethanol at 50° for 30 min. [6]. ○, untreated wool in presence and absence of detergent; □, treated wool in absence of detergent; ×, treated wool in 0.05% (wt./vol.) Nonion C detergent; ●, treated wool in 0.05% (wt./vol.) soap solution.

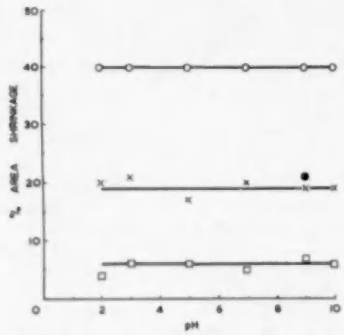

Fig. 11. Area shrinkages of ethanol-extracted wool treated with 5% (wt./vol.) sodium bisulfite at 55° for 1 hr. [6]. ○, untreated wool in presence and absence of detergent; □, treated wool in absence of detergent; ×, treated wool in 0.05% (wt./vol.) Nonion C solution; ●, treated wool in 0.05% (wt./vol.) soap solution.

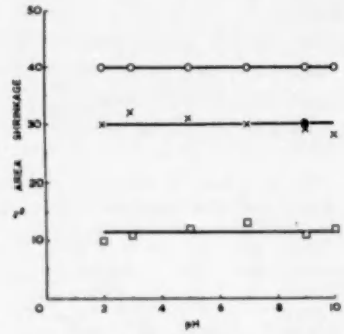

Fig. 12. Area shrinkages of wool treated with 5 vol. hydrogen peroxide, 0.2% (wt./vol.) sodium pyrophosphate, at 50° for 2 hr. followed by 1% (wt./vol.) sodium bisulfite at 50° for 1 hr. ○, untreated wool in presence and absence of detergent; □, treated wool in absence of detergent; ×, treated wool in 0.05% (wt./vol.) Nonion C solution; ●, treated wool in 0.05% (wt./vol.) soap solution.

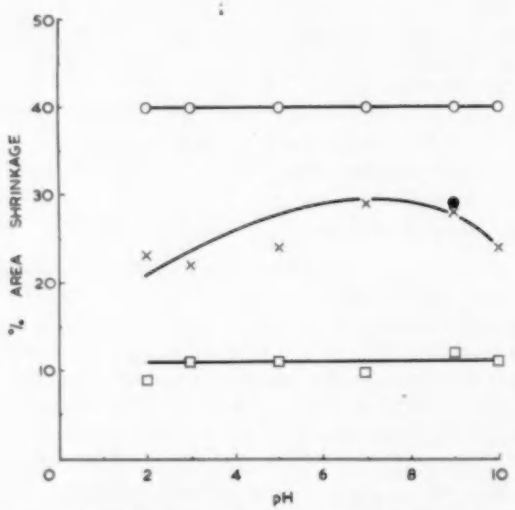

Fig. 13. Area shrinkages of wool treated with 5% (wt./vol.) sodium hydroxide in ethanol at 25° for 5 min. ○, untreated wool in presence and absence of detergent; □, treated wool in absence of detergent; ×, treated wool in 0.05% (wt./vol.) Nonion C solution; ●, treated wool in 0.05% (wt./vol.) soap solution.

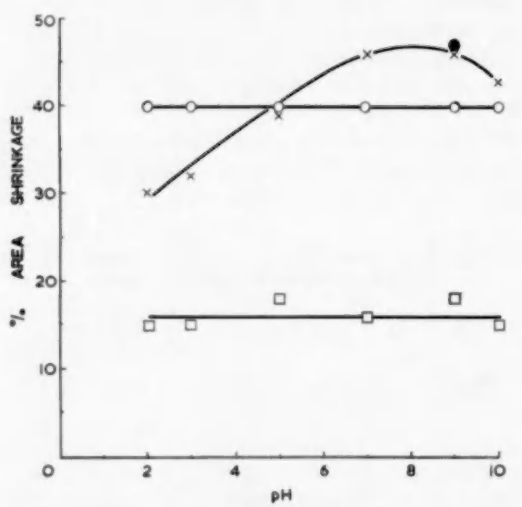

Fig. 14. Area shrinkages of wool treated with 5% (wt./vol.) sodium hydroxide in saturated sodium chloride solution at 25° for 15 min. ○, untreated wool in presence and absence of detergent; □, treated wool in absence of detergent; ×, treated wool in 0.05% (wt./vol.) Nonion C solution; ●, treated wool in 0.05% (wt./vol.) soap solution.

TABLE II. Frictional Properties

Treatment	pH of buffer solution used for friction measurement	Conc. detergent in lubricating solution	Felting curve	Frictional properties*		
				μ_2	μ_1	$\mu_2-\mu_1$
Untreated wool	9.0	0	Fig. 1	0.66 ± 0.03	0.36 ± 0.02	0.30 ± 0.04
		0.05% Nonion C		0.52 ± 0.02	0.23 ± 0.01	0.29 ± 0.02
3.5% permanganate/salt	9.0	0	Fig. 2	0.64 ± 0.03	0.40 ± 0.02	0.24 ± 0.04
		0.05% Nonion C		0.52 ± 0.02	0.30 ± 0.02	0.22 ± 0.03
1.8% acid chlorine	9.0	0	Fig. 5	0.65 ± 0.03	0.44 ± 0.02	0.21 ± 0.04
		0.05% Nonion C		0.53 ± 0.02	0.30 ± 0.02	0.23 ± 0.03
5% (wt./vol.) sodium hydroxide/salt, 25°, 15 min.	9.0	0	Fig. 14	0.68 ± 0.03	0.43 ± 0.02	0.25 ± 0.04
		0.05% Nonion C		0.65 ± 0.02	0.35 ± 0.02	0.30 ± 0.03
5% (wt./vol.) sodium bisulfite/extracted wool, 55°, 1 hr.	9.0	0	Fig. 11	0.72 ± 0.03	0.51 ± 0.02	0.21 ± 0.04
		0.05% Nonion C		0.69 ± 0.02	0.40 ± 0.02	0.29 ± 0.03
Untreated wool	3.0	0.05% Nonion C	Fig. 1	0.63 ± 0.02	0.30 ± 0.02	0.33 ± 0.03
3.5% permanganate/salt	3.0	0.05% Nonion C	Fig. 2	0.60 ± 0.03	0.37 ± 0.03	0.23 ± 0.04
5% (wt./vol.) sodium hydroxide/salt, 25°, 15 min.	3.0	0.05% Nonion C	Fig. 14	0.85 ± 0.03	0.60 ± 0.03	0.25 ± 0.04
5% (wt./vol.) sodium bisulfite/extracted wool, 55°, 1 hr.	3.0	0.05% Nonion C	Fig. 11	0.87 ± 0.03	0.63 ± 0.03	0.24 ± 0.04

* Figures given are means and standard deviations of measurements on at least 10 fibers.

ous bisulfite on extracted wool are shown in Table II), the frictional coefficients are again lowered by the addition of detergent to the solution, but here the frictional difference ($\mu_2 - \mu_1$) is increased by the detergent. (For sodium hydroxide/salt, the difference in $\mu_2 - \mu_1$ is significant at the 5%, but not the 1%, level). With both the treatments given, frictional coefficients are higher and frictional differences lower at pH 3 than at pH 9. (The differences in $\mu_2 - \mu_1$ are significant at the 5%, but not the 1% level.)

Discussion

Under our conditions, the rate of felting of untreated fabric decreased with increasing pH over the range pH 2 to 10, and the rate of felting at all pH values was increased by addition of detergent to the felting liquor. With untreated and all treated samples, rate of felting was the same in 0.05% Nonion C solution in a pH 9 buffer and in 0.05% soap solution at about the same pH.

The treatments studied fall into three groups. Similar shrinkage–pH curves, showing increased felting with decreasing pH, were obtained with seven industrially used oxidation. The rates of felting of the oxidized samples, relative to untreated samples, were unchanged by the addition of detergent to the felting liquor.

Another group consists of treatments (ethanolic thioglycolate, aqueous bisulfite on extracted wool, and peroxide followed by bisulfite) giving good shrink resistance which does not vary with pH when tested in the absence of detergents. In the presence of detergents, however, felting is much more rapid but again does not vary, relative to untreated wool, with pH.

Sodium hydroxide treatments in ethanol or saturated salt solution form a third group, giving a shrink-resistant effect not varying with pH in the absence of detergent but felting more rapidly when detergent is added, the shrinkage–pH curves then showing maxima at pH 7–9.

It is generally considered that felting behavior of wool can be explained in terms of frictional and elastic properties of the fibers. On this basis, a simple relation has been established for felting of hypochlorite-treated wool in different media [18],

but it is doubtful whether this could be applied to felting in aqueous solutions of fabric treated by a variety of reagents [10]. Other facts have been presented which also do not fit the simple scheme [6]. Qualitatively, the concept that weakening of fiber causes greater felting [21], together with the logical supposition that wool will felt faster the lower the frictional coefficients and the higher the frictional difference, can be used in an argument to try to explain some of the results given in this paper.

The following short discussion is quite general and speculative but is based on commonly accepted ideas on felting. The only physical property, other than felting, we have measured is friction against a keratin rod. It should be borne in mind that different results may be obtained in the future by fiber–fiber friction measurements, e.g., by the fiber twist technique [7]. Detailed information on mechanical properties of fibers and fabrics which may play an important role in felting is not yet available to allow the discussion to be more specific.

In the case of untreated wool, it could be maintained that detergents increase the felting rate at pH 9 by lowering the frictional coefficients without changing the frictional difference. The increasing felting rate with decreasing pH may be due to an increase in frictional difference combined with decreasing resistance to extension [19]. These factors would have to be considered to more than compensate for the higher frictional coefficients in acid solution, in order to account for the observed increase in felting rate.

The effect of detergent at pH 9 on wool treated with oxidizing agents used industrially is similar to that on untreated wool, a lowering of the frictional coefficients taking place while the frictional difference remains unaltered. The relative rate of felting, compared with untreated wool, should not therefore be changed by the detergent.

With treatments where detergent increases the relative felting rate, the detergent also lowers the frictional coefficients and increases the frictional difference; this is in agreement with the argument. In the presence of detergent at pH 9, the actual frictional coefficients are still very much higher than those of untreated wool, and this alone should give a low rate of felting. However, sodium hydroxide/salt-treated wool felts more rapidly than untreated wool (Figure 14) and bisulfite-treated extracted wool still felts much more than might have been expected (Figure 11) for such a large increase in frictional coefficients. It would have to be argued, then, that increased felting due to weakening of fibers during treatment is masked, in the absence of detergent, by the frictional properties which cause a decreased felting. When detergent is present, the frictional properties are changed sufficiently to allow the fiber-weakening to play a more significant part in the case of bisulfite-treated extracted wool, and to play the major part in the felting of sodium hydroxide/salt-treated wool.

The frictional and elastic properties do not appear even qualitatively to explain the effect of pH on the relative felting rate of the treated fabrics. For example, the permanganate/salt treated-fibers would have to be weakened relatively much more than untreated fibers with decreasing pH to give the increased relative felting rate with little relative change in frictional properties. The bisulfite-treated, extracted fibers would also have to be weakened more than untreated ones at low pH, since the frictional properties at low pH should cause a lower rate of felting relative to untreated, compared with the felting properties in alkaline solution.

It is difficult to visualize why, say, the elastic properties of 3.5% permanganate/salt-treated fibers should be affected so differently than are untreated fibers as the pH of the felting solution is lowered. The only known change is oxidation of about 4% of the cystine. It might well be that a much more profound change has taken place in the cuticle than in the whole fiber and that this could cause the very different relative felting behavior with pH of treated wool compared with untreated wool.

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Shrink-Resist Treatment and Laundering of Wool Fabrics

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Abstract

Commercial wool fabrics may shrink on wetting, due to the relaxation of strains imparted in processing, in washing, due to felting, an inherent property of wool fiber, and in the tumble drying of wet fabrics, due to a reversible relaxation of structure. The common industrial methods for reducing felting by chemical reactions are described. Many factors affect the rates of these reactions, and it is essential to control the processes adequately if shrink resistance of the required level is to be consistently obtained. The types of machines used for treatment and the importance of efficient scouring and of obtaining level treatments are discussed.

The properties of wool treated by the various methods are described. It is often better to dye or bleach after shrink-resist treatment, but it is shown that prolonged boiling must be avoided since shrink resistance and strength may be adversely affected and relatively high weight losses obtained.

Machine laundering of woven and knitted fabrics is discussed in terms of type of machine action, load in the machine, fabric structure, and degree of shrink-resist treatment. All these factors must be considered simultaneously in the commercial production and sale of washable wool goods.

Introduction

This paper describes the principles of some common industrial methods of making wool shrink-resistant by chemical treatment and the behavior of wool fabrics, both untreated and treated, in various laundering procedures.

There are many discussions on the shrinkage of wool fabrics (see e.g. [26] and references there). In general shrinkage has been divided into the two main classifications of relaxation and felting. ("Consolidation shrinkage" [12] is ill-defined and may be simply a type of relaxation of loose structures. The

"consolidation" which takes place on tumble drying [13], for example, depends on fabric structure [13] and is a relaxation of some type which can be reversed by re-wetting. The distinction is important because of the completely different characteristics of the different types of shrinkage.

If goods are dried completely free of tension after their final wet treatment, instead, as is common practice, of being stretched during drying, they have little or no simple relaxation shrinkage [13]. Since the stable, dry dimensions are lower than wet dimensions, this desirable result is difficult to obtain with the use of multi-layer tentering machines, and fabrics dried in the usual way may show considerable relaxation shrinkage when simply wet out. The shrinkage is due to release of the strains imparted to the yarns in the fabric while stretched during drying. Washable fabrics should have relaxation shrinkages at the minimum possible and this can be attained either by tensionless drying or by sponging the fabrics after finishing in the mill. Final pressing or other procedures which cause warp extension should be avoided.

Many knitted goods are not wet processed after knitting. In this case, a single relaxation treatment of steaming or wetting usually brings the fabric to near its true stable dimensions, at which it should be cut or pressed without undue stretching at any later stage. Relaxation shrinkage has been a problem for at least 500 years [27] and remains so, in some cases at least, simply because of convenience.

On the other hand, felting shrinkage is due to fiber movements and entanglements resulting from the application of undirected mechanical forces [24], as in laundering. Structure plays a part in the rate of felting of wool fabrics; the lower the yarn twist, the looser the weave or knit, and the higher the applied forces, the greater is the rate of felting [4, 5, 12, 13, 14, 17, 35]. Very tight structures can be laundered in the mildest types of washing machine with little felting [3, 4, 13], but there are many structures which require a shrink-resist treatment before they can be washed satisfactorily even under the gentlest conditions. Relatively few treatments (see, e.g., [26]) have reached industrial importance and most of these involve the use of free chlorine or hypochlorite. The common industrial processes generally can produce satisfactorily shrink-resistant wool with no significant adverse effects on the desirable properties of the fiber (see, e.g., [22]), although wool chlorinated either dry or in acid solution normally requires a softening treatment to be completely acceptable.

The forces involved in laundering by various methods are quite different [6, 23], and a given fabric, either shrink-resistant or untreated, can behave differently in different washing machines or in the same machine at different speeds or loads [23]. The three main factors affecting rate of felting must therefore be considered simultaneously when producing and selling wool goods. These are fabric structure, degree of shrink-resist treatment, and laundering procedure to be used.

Common Industrial Chemical Shrink-Resist Treatments

The essential technical details of some processes are freely available [e.g., 9] but others must be obtained from the licensors of trade marks or patented processes. Resin treatments are still not in wide use. Their apparent simplicity, padding followed by drying and curing, is often complicated by essential pretreatments, special equipment for curing, and a need for thorough washing off and softening.

Acid Chlorinations

In their simplest form, acid chlorinations involve the slow addition of sodium hypochlorite solution to an acid liquor in which the wool has been thoroughly equilibrated [28]. Hydrochloric acid was the first acid in common use and formic acid is used in the Negafel process [8]. These reactions are very rapid and are only level and satisfactory if the temperature is kept to the practical minimum. Methods of slowing the reaction are available. Some use auxiliaries added to the bath before the hypochlorite, for example Melafix (Ciba) or Chloregal D (Geigy), but it is important not to slow the reaction to such an extent that the temperature has to be raised, since handle and color can then deteriorate rapidly. Another method of controlling the rate is to add an oxidizing agent, e.g., potassium bromate, to an acid solution of a chloride [18, 22]. Chlorine can be liberated at any desired rate by adjusting the concentrations of acid, chloride, and oxidizing agent. The higher the concentration of sodium chloride, the less chlorine is needed for a given degree of shrink resistance and the slower is the rate of reaction of chlorine with wool [16], both factors being of advantage for industrial processing.

The modifications help considerably in producing level treatments, and consequently ease of dyeing afterwards, but we find that the other properties of treated wool, including the harshened handle, depend mostly on the amount of chlorine which has reacted.

The methods can be used for continuous treatments [20, 22] as well as for the more common batch processes.

Neutral Chlorinations

In general, so-called "neutral" chlorinations are carried out in the pH range 6–9. In the simplest form, hypochlorite is added to a solution buffered to maintain the pH of the wool and liquor at a desired figure, usually about 8. Under these conditions, more active chlorine is needed for a given degree of shrink resistance, yellowing is much more pronounced, and the reaction with wool is slower than in acid solution [7, 11, 15]. A recent development is the use of a resin pretreatment to increase the efficiency of the method [25]. The method is also used continuously as the Harriset process [15] and probably by mills under their own trade marks.

It is convenient to classify here processes which use hypochlorite in this approximate pH range in combination with permanganate (Dylan Z process [33]) or peracetic acid [10]. In these methods, the combination of reagents gives a much higher degree of shrinkage resistance than either reagent alone at the given concentrations.

The properties of wool treated by the different methods, for a given degree of shrink resistance, are the same for all practical purposes [11, 22, 25].

Dry Chlorination

This needs a special plant for the treatment with free chlorine gas, and special organization and equipment is required to ensure that the wool has the correct moisture content before treatment [29, 32].

Permonosulfuric Acid Followed by Reduction

The controlled treatment with a sulfite-bisulfite solution, after a simple oxidation with the permonosulfuric acid [34], is an essential step in the development of the shrink resistance. The process can be applied in continuous treatments at room temperature in a conventional backwash unit with one or two added bowls [34] as well as by the usual batch methods. These treatments are known as the Dylan X process or Dylanize.

Permanganate/Salt

The efficiency of neutral permanganate as a shrinkproofing reagent can be increased either by addition of hypochlorite to the liquor [33] or by saturating the liquor with sodium chloride [21, 22]. As a batch process, the permanganate/salt method has a great advantage over the processes described above in simplicity of control. No unstable reagent requiring regular analysis is used, and both the extent and the levelness of treatment can be followed visually from the pink color of the solution and the deposition of manganese dioxide on the wool.

The continuous treatment with permanganate/salt [22], however, requires the careful control of concentration and temperature generally associated with continuous processing.

Industrial Application of Shrink-Resist Treatments

For the best and most consistent results, the wool should be clean and conditions of treatment chosen to ensure that all fibers are uniformly treated. Level treatment is necessary to obtain maximum shrink resistance for a given amount of shrinkproofing reagent used, since a relatively small proportion of untreated fibers can have a large effect on the rate of felting [2]. In general, we have found that for wool treated evenly by the permanganate/salt process, a given structure has the same degree of shrink resistance whether fibers are treated in the sliver, at the yarn stage, or after the fabric has been constructed. It is important to note that "undertreated" fibers may behave almost like untreated and that "levelling" by gilling, or combing unlevel, treated sliver does not always improve the shrink resistance of the fiber assembly as a whole. Level treatment is also important to ensure level dyeing after treatment, although the various treatments do affect rates of dyeing quite differently [19, 22, 31].

In some treatments it is possible to obtain satisfactory results with appreciable amounts of certain oils and impurities on the wool, but in general it is best to clean the wool as thoroughly as practicable before treatment. In some cases, for example knitted goods containing 3–6% oil, some treatments impart no shrink resistance at all if the goods are not prescoured. Scouring with built, nonionic detergents is ideal, as even with the mildest of agitation in batch

TABLE I. Effect of Prior Low Level Shrink-Resist Treatment on a Subsequent Re-Treatment

Initial treatment on normal wool	Re-treatment	Area shrinkage, %
—	—	40
3% permanganate/salt	—	18
4% permanganate/salt	—	8
5% permanganate/salt	—	3
3% permanganate/salt	1% permanganate/salt	18
3% permanganate/salt	2% permanganate/salt	14
3% permanganate/salt	3% permanganate/salt	11
3% permanganate/salt	4% permanganate/salt	6
3% permanganate/salt	5% permanganate/salt	3

processing (e.g., slow paddle machines) or in the shortest of times in continuous processing (e.g., 10 sec. for backwashing sliver), the oil and grease content can be reduced to 0.1–0.2%. Best scouring conditions vary because the concentration of active ingredient varies in the commercial detergents but, in general, 0.05–0.2% (wt./vol.) of the nonionic detergent plus 0.1–0.5% (wt./vol.) of common salt, sodium sulfate, or sodium bicarbonate at 40°–50° C. for 15 min. is satisfactory for batch processing. For a conventional backwash, 0.2% detergent plus 0.5% salt will often scour about 500 lb. wool before topping up or replacement of the liquor is necessary.

The times of reaction of a given amount of shrink-proofing reagent with the wool can vary considerably depending on a large number of factors. This means that it is unlikely that consistent results will be obtained industrially if one set of "standard" conditions is taken from trade publications and used indiscriminately. Factors affecting the rate of reaction include the fiber type, the structure of the fiber assembly, the type of machine used (which determines the degree of agitation or rate of flow of liquor through the wool), the conditions used for dyeing if the wool has been dyed, the presence of oils and impurities on the wool, the wetting agent used in the liquor, the liquor:goods ratio, the temperature, in many cases the pH of the liquor, and in gaseous treatments, the moisture content of the wool. Many, but not all, of these variables can be kept constant in particular applications, and it is necessary to control the processes analytically to establish that each batch has received the required degree of treatment. This means, of course, that processes involving the use of unstable reagents like hypochlorite or peracetic acid, also require regular initial analyses of the strength of the stock solutions.

Experimental runs should be done on each fabric to be treated to establish the concentrations of reagents required for the given structure. Relatively "loose" fabrics may need more reagent than "normal" ones, while very tight fabrics may require less. Also, more effective agitation than usual may be needed for the reagents to penetrate very tight structures adequately.

We find that for level, satisfactory wet treatments, the conditions should be adjusted to give a half-life of reaction (i.e., the time taken for half the initial quantity of reagent added to react with the wool) of 8–10 min., preferably 10 min. For gaseous chlorination, the moisture content needs to be adjusted to about 6–8% [32] before treatment.

Wet treatments generally can be carried out in conventional dyehouse equipment. A qualification is that the essentially inefficient open-can dyeing machines used for tops often do not provide satisfactory flow of liquor through the tops for level treatments. Some modification of the pumping system or the density of the tops to be treated may be necessary, and it is much better industrially to use top dyeing machines equipped with spindles which pass through the centers of the tops, or better, pressure dyeing machines which are normally built with far more efficient pumps. It has been found, nevertheless, that mildly uneven treatments in open machines can still give a reasonably high degree of shrink resistance when the wool has been converted to relatively tight fabrics. However, unlevel treatments of top suffer from the further disadvantage that "overtreated" fibers will break more easily during later gilling, combing, and finishing, and undue weight loss may occur.

Re-Treatment of Inadequately Treated Wool

If a fabric has been given a shrink-resist treatment at a level too low for a given purpose, it may sometimes be necessary to re-treat the fabric with that concentration of reagent which would have been required had it not been previously treated. This is illustrated by the results in Table I. Fabric, treated with 3% permanganate/salt to give 18% area shrinkage in a standard washing test, was then re-treated with various concentrations of permanganate. Re-treatment with 4–5% permanganate was necessary to give an area shrinkage equivalent to that obtained with 4–5% permanganate on the normal untreated wool. We have observed many

times that fabrics treated industrially in unknown ways (said to be "chlorinated"), but which still were not as shrink-resistant as required, needed re-treatment with 6–8% permanganate to give the same shrink resistance that could be obtained on an identical untreated structure with 4–5% permanganate/salt.

Effect of Prior Boiling of Wool on Shrink-Resist Treatments

If boiling during dyeing before treatment is carried out for an abnormally long time in a strongly acid solution, it may be necessary to increase the amount of shrinkproofing reagent to achieve the effect required (Lipson, unpublished, for ethanolic potassium hydroxide treatment; [21]). This means that further trials should be carried out if it is known or suspected that a given fabric has been made from waste wool or redyed wool. However, boiling for the normal time has little effect, and even longer than usual times in weakly acid or neutral solutions usually has no significant effect on the degree of shrink resistance obtained under given conditions [21].

The low strength loss often obtained when undyed wool is made shrink-resistant (Table II) is rarely any greater when dyed worsted fabrics are treated. We have found strength losses greater than those given for the unboiled samples in Table II, however, when treating some, initially much weaker, dyed woolen fabrics.

Dyed wool loses slightly more weight than undyed during shrink-resist treatments (Table III). In general, weight losses of 0.5–1.0% are to be expected when dyed wool is treated with the reagents studied in this work.

Effect of Boiling, Aqueous Solutions on Shrink-Resistant Wool

The effect on fabric properties of dyeing after shrink-resist treatment is of considerable practical importance, and some results with a plain weave worsted fabric of Merino 64's wool are given in Table II. The results should apply qualitatively to other fabrics but are probably only quantitatively significant for this one fabric.

The boiling of normal wool reduces its strength, but the loss in strength is significantly greater if the wool has first been given a shrink-resist treatment (Table II). Boiling in strongly acid solutions weakened this fabric more than boiling in neutral or slightly acid solutions. Acid chlorinated fabrics are always stronger than other shrink-resistant fabrics given the same aftertreatments (Table II). Boiling in the presence of dyes gave exactly the same results as those reported in Table II.

Wool treated with permanganate/salt, permonosulfuric acid, or peracetic acid/hypochlorite tends to lose some shrink resistance when boiled for lengthy periods, especially in acid solutions (Table II). However, the effect of normal dyeing conditions appears to be of little practical significance.

The oxidations taking place during shrink-resist treatment increase the alkali solubility and soap solubility of wool [21, 22] but not the solubility in solutions of nonionic detergents. The results in Table III show that the solubility in boiling aqueous solutions, such as used for dyeing, is also significantly increased by the treatments. The weight losses shown were determined in blank dyebaths containing 3% (on the weight of wool) ammonium sulfate, since these are the simplest to determine and have made a useful damage test. Weight losses in acid solutions are more difficult to determine, since the acid sorbed by the wool must be neutralized after boiling and the wool then washed free from salts. Weight losses in acid solutions commonly used for dyeing, and in all typical solutions with and without dye in the liquors, are close to those shown in Table III. Actual wool losses of 1–2% during shrink-resist treatments and subsequent dyeing are to be expected. Determinations made in mills varied considerably. Lower weight losses were sometimes found, probably due to sorbed acid and salts. Higher weight losses were found when dyeing was carried out for longer times than normal.

Properties of Shrink-Resistant Wool

Acid and gaseous chlorinations increase the rate of dyeing of wool very markedly [e.g., 19, 22, 31] so that even moderately unlevel treatments can cause unlevel dyeings with most dyes. The increased rate of dyeing due to treatment with permanganate/salt is quite low, and with permanganate/hypochlorite not very much higher [22]. Peracetic/hypochlorite, simple neutral chlorination, and permonosulfuric acid often decrease the rate of dyeing [19, 22, 31]. Treatments with all these reagents can be slightly unlevel without causing much trouble in dyeing, but in general, the aim should always be to obtain

TABLE II. Effect of Boiling, Blank

Blank dyeing conditions—concentrations expressed as % on weight of wool; liquor:wool ratio of 30:1	4% permanganate/salt			2% acid chlorine*			2% permonosulfuric acid		
	Area shrinkage, %	Bursting strength		Area shrinkage, %	Bursting strength		Area shrinkage, %	Bursting strength	
		Conditioned‡	Wet		Conditioned‡	Wet		Conditioned‡	Wet
—	5	108	81	5	114	86	4	111	82
16% H_2SO_4, 15% Na_2SO_4, boiled 2 hr.	9	96	67	5	99	70	8	94	65
6% H_2SO_4, 15% Na_2SO_4, boiled 2 hr.	7	100	70	5	103	72	4	99	70
4% $(NH_4)_2SO_4$, boiled 2 hr.	6	105	70	6	107	72	4	104	70
6% acetic acid, boiled 2 hr.	6	105	71	5	107	72	6	105	71
6% acetic acid, 15% Na_2SO_4, boiled 30 min.; 1% H_2SO_4 added, boiled 30 min.; 5% $K_2Cr_2O_7$ added, boiled 45 min.	9	102	69	3	104	72	8	103	69

* Treatment by the bromate/saturated salt method [22].
† "Chlorine" means the quantity of active chlorine in the hypochlorite added.
‡ 20° C., 65% RH

the maximum degree of levelness in the shrink-resist treatment.

For the accurate production of shades, especially pastels and bright colors, it may be better to dye after the shrink-resist treatment, since many dyes are changed by the oxidations (see manufacturers' trade information for details). It should always be borne in mind, however, that if boiling is prolonged in order to match a shade or to level the dye, there can be an adverse effect on both shrink resistance and strength, and weight losses can be relatively high.

The washfastness of dyes of good fastness is only slightly lower on shrink-resistant wool [1, 19, 31] than on normal wool. Information about specific dyes can be obtained from dye manufacturers' trade circulars.

It is usually better to bleach after the shrink-resist treatment rather than before, since these treatments may yellow the bleached wool somewhat (Table IV). Peroxide treatment may reduce the degree of shrink resistance of the treated wool, but a further bisulfite treatment completely restores the shrink resistance and at the same time improves the bleach (Table V).

A summary of many other properties of the treated wools has been given previously [22], as have also the concentrations of the different reagents required to produce an equivalent degree of shrink resistance [23]. All the processes described are capable of giving similar, satisfactory results. A high degree of shrink resistance is easily obtained with quite low direct chemical and labor costs; the color and handle of wool are unaffected (provided acid or dry chlorinated wool is softened); treated goods can generally be dyed level (provided, in the case of acid chlorination, that a modification to slow the reaction is used); and the washfastness of dyes of good fastness is adequate on the treated wool.

The choice of process and the stage of processing at which treatment is to be carried out usually does not depend on any differences in properties of the treated wool. The choice depends on simplicity of

TABLE III. Loss in Weight of Wool Resulting from Shrink-Resist Treatment and Boiling *

Shrinkresist treatment, % reagent conc. on wt. wool	Weight loss on shrink-resist treatment, %	Weight loss, %, after	
		Boiling followed by shrink-resist treatment	Shrink-resist treatment followed by boiling
—	—	—	(0.3)
4% permanganate/salt	0.5	0.8	1.5
2% acid chlorine	0.6	1.0	1.0
2% permonosulfuric acid	0.2	0.5	1.1
2% permanganate +2% chlorine†	0.9	1.1	1.5
1% peracetic acid +3% chlorine†	0	0.4	2.2
1.5% gaseous chlorine	0	0.3	1.6

* Fabrics were boiled for 1 hr. in 30 times their weight of water containing 3% ammonium sulfate (on the wt. wool).
† "Chlorine" means the quantity of active chlorine in the hypochlorite solution added.

Dyebaths on Shrink-Resistant Wool

2% permanganate + 2% chlorine†			1.5% gaseous chlorine			1% peracetic acid + 3% chlorine†			No shrink-resist treatment		
Area shrinkage, %	Bursting strength		Area shrinkage, %	Bursting strength		Area shrinkage, %	Bursting strength		Area shrinkage, %	Bursting strength	
	Conditioned‡	Wet		Conditioned‡	Wet		Conditioned‡	Wet		Conditioned‡	Wet
3	108	79	6	112	83	5	109	81	40	114	86
4	97	67	8	91	59	8	96	66	44	101	71
4	98	69	5	95	66	6	101	71	43	108	75
3	104	68	7	101	70	6	105	70	42	108	75
4	105	69	7	102	71	7	106	70	43	109	75
3	103	69	7	100	70	9	104	69	42	112	76

technique, cost of any new equipment required, and possible indirect costs: for example, decomposition of stock solutions of unstable reagents; maintaining the close analytical or temperature control required by some processes (otherwise there is an indirect cost arising from inconsistent results on different batches); faulty dyeings, which occur most frequently with the processes which tend to yellow the wool or change the rate of dyeing most; or payment of royalties for patented processes.

The problem of weight loss is more important the earlier the stage at which shrink-resist treatment is carried out. Weight loss is small and unimportant when dyed garments are treated and, in most cases, can be ignored when dyed pieces are treated. Weight losses during treatment of dyed loose wool, tops, or yarn are always quite low but will still have to be allowed for in costing if the treated wool is to be sold by weight.

TABLE IV. Effect of Permanganate/Salt Shrink-Resist Treatment on Peroxide Bleached Wool

Peroxide treatment	Shrink-resist treatment	Reflectance	Area shrinkage, %
(untreated)		(53)	(40)
2 vol., 0.2% pyrophosphate, 2 hr., 50° C.	—	64	34
2 vol., 0.2% pyrophosphate, 2 hr., 50° C.	4% permanganate/salt	60	6
5 vol., 0.2% pyrophosphate, 2 hr., 50° C.	—	67	33
5 vol., 0.2% pyrophosphate, 2 hr., 50° C.	4% permanganate/salt	61	6

Where dyeing follows shrinkproofing, it is essential that both the quantity of shrink-resist reagent and the time consumed in dyeing be kept to a minimum to avoid relatively high strength and weight losses.

Machine Washing of Wool Fabrics

The variables in machine washing of wool fabrics considered here are: the type of machine action; the load of goods in the machine (i.e., the liquor : goods ratio of the wash); the structure of the fabric (including yarn structure); and the degree of treatment given to the fabric to achieve shrink resistance. Variables which play a part in rate of felting, but which are normally constant within reasonable limits, are the temperature of the washing liquor and the concentration of soap or detergent.

TABLE V. Effect of Bleaching on Shrink-Resistant Wool (Treated with 4% Permanganate/Salt)

Peroxide treatment	Bisulfite treatment	Reflectance	Area shrinkage, %
(untreated wool)	—	(53)	(40)
(shrink-resist treatment only)	—	53	5
—	1% wt./vol., 50° C., 30 min.	55	3
2 vol., 0.2% pyrophosphate, 2 hr., 50° C.	—	62	11
2 vol., 0.2% pyrophosphate, 2 hr., 50° C.	1% wt./vol., 50° C., 30 min.	65	4
4 vol., 0.2% pyrophosphate, 2 hr., 50° C.	—	64	12
4 vol., 0.2% pyrophosphate, 2 hr., 50° C.	1% wt./vol., 50° C., 30 min.	67	4

Types of Machine Action

Agitator action. This results in a gentle motion of goods both round and up and down through the liquor and is brought about in the following general ways: (a) by reversing, centrally-mounted vanes, (b) by a vertical reciprocating motion of a central cylinder with protruding rings, (c) by agitation of the bowl itself, (d) by the rapid eccentric movement of a complete central unit of vanes.

In any of these systems, it is common to find at least two machine speeds, sometimes three. The detergent action is adequate even though the movement of goods may not appear to be very rapid. The rate of felting of untreated wool fabrics is least in these types of machine [23], especially when the lowest available speed is used. There is, however, a critical load in the machines, usually in the range 4–6 lb., above which the rate of felting may be increased significantly [3, 23]. The gentle motion of the goods is then changed to a buffeting against other goods or the sides of the bowl, and the forces applied to the fabrics are thereby increased.

Rotating drum action. In these machines, a horizontally mounted, cylindrical drum carrying the goods rotates through the liquor. The milling action, as the goods are lifted and dropped back, results in relatively high forces being applied to the fabric. Load is not particularly important, but the speed of the machine and soap concentration are, and the rate of felting may be much higher than in agitator machines [23].

Impeller action. The liquor and goods are moved by a small impeller fitted into the side of the washing bowl. This results in a severe action when the impeller is rotating at its normal speed of about 700 rev./min., and again load plays only a small part in the rate of felting. The rate of felting is decreased considerably by slowing the speed to about 475 rev./min. [23].

Washing of Untreated Fabrics

The rate of felting of untreated wool fabrics shows great variation in these different machines [23]. For a loosely woven fabric, the initial rates of felting have been shown to vary from as low as 0.2% area shrinkage/min. in a slow-speed agitator wash to 2.4% area shrinkage/min. in a rotating drum machine [23]. Machines can be conveniently classified as "gentle," which includes agitator types at low speed, or "severe," which may include both rotating drum and impeller types [23].

Structure plays a large part in the rate of felting of fabrics [4, 5, 12, 13, 14, 17, 35]. In untreated woven fabrics, initial rates of shrinkage can vary at least from 0.3% to 2.4% area shrinkage/min. in a rotating drum machine; while in knitted fabrics, the range can be at least from 0.7% to 3.7% area shrinkage/min. [23]. Fabric structures can be expressed in terms of a compactness ratio for wovens [4] or in terms of stitch length for knitteds [30].

Washing of Shrink-Resistant Fabrics

Industrial shrink-resist treatments are capable of making any fabric nonfelting in any machine. However, other factors besides the nonfelting property need to be considered, and in some cases complete nonfelting is obtainable only with disadvantageous side effects, such as loss of fiber strength, loss of desired handle, loss of appearance through washing, and stretching of the treated fabric when given mild, rather than severe laundering.

Woven fabrics. In our experience, many woven fabrics can be made "machine washable" (e.g., [13]) in the broad meaning of the term, without adverse side effects. Studies, on an industrial scale, have been made of treated blankets which can be laundered at least 250 times by boiling in hospital laundry washing machines, and of treated fabrics manufactured into shirts, blouses, dresses, skirts, trousers, and children's schoolwear. All the treatments discussed can give similar results.

Knitted fabrics. Treatment needs to be at as low a level as possible to retain the soft handle associated with knitted woolens and also to restrict the drop in fiber strength to a minimum. However, treatment must be adequate to produce fabrics which do not felt under the recommended washing conditions.

Many knitted fabrics, for example, relatively tight plain knits (stitch length 0.45–0.55 cm. [23]) or double knit jerseys, felt at rates comparable to wovens and can easily be treated to perform equally well in machine washing. Many others, for example, loose plain knits (stitch length 0.8–0.9 cm.) or bulky knits, felt much more rapidly and when given a "normal" treatment may still be quite unsatisfactory if washed in a severe machine [23]. It is possible to have a fabric appear completely shrinkproofed if washed at slow speeds in a gentle machine

but still show a large felting shrinkage after, say, 30–60 min. in a severe machine [23]. Loose fabrics, if given a high degree of treatment, will be satisfactory in severe washes but may then be unsatisfactory in gentle or very short washes due to stretching of the loose stitch structure.

Summarizing, woven and certain knitted wool fabrics, if treated correctly, can be called machine-washable. Loose knit, treated fabrics, however, must have the type and speed of machine specified if they are to be machine washed.

In general, garments which are machine-washable in modern home equipment can be made from treated all-wool fabrics. Machine washability of garments (made from synthetic as well as natural fibers) is often limited by the washfastness of the dyes used and by construction factors—that is, the type and strength of seams, the sewing threads, the skill and consistency of the sewing machinist, the maintenance of appearance of the fabric structure or shape of the garment—rather than by shrinkage or abrasion of the fabric during laundering. In particular, machine washing of fabrics containing loosely twisted yarns can rapidly raise a nap, with consequent loss of appearance.

Acknowledgments

I should like to thank Dr. M. Lipson and Dr. A. J. Farnworth for helpful discussions and Mr. H. D. Feldtman for assistance with the industrial-scale shrinkproofing trials and other experimental work carried out during this study.

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The Kinetics of the Absorption and Desorption of Single Ions by Keratin Fibers

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Abstract

Rates of absorption, desorption, and exchange of both cation and anion by hair in Na_2SO_4 solutions have been measured using a radioactive tracer technique. These are compared with results obtained previously using alkali halides, Na_2SO_4 and H_2SO_4.

Introduction

In the course of a study of the behavior of ions within keratin fibers (as represented by human hair), several papers have been published which contain results on the rates of absorption or desorption of ions by fibers immersed in aqueous solutions [1, 4, 6–8]. The measurements were made using radioactive tracers, and three processes were followed: absorption, desorption, and exchange. The process called exchange involves diffusion in the absence of a chemical potential gradient for the material involved. For example, a fiber is brought to equilibrium with a solution containing a radioactive ion and then transferred to a solution of the same chemical composition and concentration which does not contain a radioactive ion. The radioactive ions diffuse out of the fiber but there is no gradient of chemical potential and no net transfer of matter. All of the experiments were characterized by an essentially infinite bath-to-fiber ratio, so that the concentration of the treating bath remained unchanged. The fibers used were preswollen in water vapor.

In addition to the results already reported, some further measurements have been made on the uptake of Na^+ and $SO_4^=$ ions from Na_2SO_4 solutions. In these measurements greater care was taken in adjusting the initial pH of the treating solution and in following the pH of the solution during the absorption or desorption to make sure that no change occurred. Greater care was also used in adjusting and controlling the pH of the wash liquid. Otherwise the technique used was the same as that reported in the earlier papers [1, 4, 6, 7] (for complete details see [5]).

The rate measurements have been brought together in this paper and augmented by the more carefully controlled measurements made using Na_2SO_4 solutions. In this way certain trends and modes of behavior can be picked out and discussed.

Results

Results for all of the diffusion processes that have been studied are collected in Table I. The values of the parameters controlled, the times of half completion of the diffusion process, and the apparent diffusion coefficients are given.

An accurate estimate of the probable error is not possible. There are three different sources of error: the measurement of the amount of a given ion in a sample, the determination of an empirical curve for the progress of the diffusion process, and the estimation of a diffusion coefficient. On top of this, the reliabilities of the various examples in Table I differ, both because the suitability of the techniques improved with time and because in some cases the primary purpose of the measurements was to find a suitable time interval to ensure equilibrium, so that complete rate curves were not obtained.

Analyses of the errors involved in the tracer experiments and the reproducibility of repetitive measurements of equilibrium results are given in the references cited and fall in the range of 5–8%. For various reasons the range becomes 10–12% for the rate measurements, where fewer fibers were used for any one point. In most cases, and especially in

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those involving Na_2SO_4 where the pH was controlled in the sorption baths (pH_S) and in the desorption baths (pH_D), an empirical curve could be drawn which passed within 10% of each of the points. Some of these curves are shown in Figures 1 and 2. The value of $t_{\frac{1}{2}}$ may not be quite as accurate as the fit of the empirical curve, both because the curve may be changing rapidly (on a semilog plot) at $t_{\frac{1}{2}}$ and because in some cases (some alkali halides) results are missing in the immediate vicinity of $t_{\frac{1}{2}}$. In Table II the half-times of absorption and desorption are compared where possible.

The apparent diffusion coefficients have been calculated using the equation appropriate for a cylinder of fixed radius in an infinite bath and with a diffusion coefficient independent of concentration. In general the fit of the theoretical curves to the experimental points is poor so that the apparent diffusion coefficient is more valuable as an index of the relative rate of the diffusion process involved than as a means of quantitatively characterizing that process.

Discussion

The first feature noticeable in Tables I and II is the wide range of half-times observed, covering four decades of time. This range probably reflects the complexity of processes being observed and the variety of factors which may be rate determining. The entries in Table II can be divided into three categories: processes where desorption is markedly faster than absorption, processes where the two proceed at about the same rate, and one case where desorption is slower than absorption. To analyze the results further some discussion of theoretical background is helpful.

If Fick's law is applied in the cases of diffusion from an infinite bath into a cylindrical fiber of fixed size and from the fiber into a bath of pure solvent, the results show [3] that the half-times of absorption and desorption should be equal. Three possible reasons for departures from Fick's law, which could be considered applicable for various reasons in the cases considered here, are immobilization of part of the diffusant by adsorption on sites, the presence of an outer layer or skin of different permeability leading to a compound diffusion process, and a concentration-dependent diffusion coefficient.

The case of combined adsorption and diffusion has been considered by various workers (see [3]). It can be shown [3, 5] that, if the amount adsorbed is a linear function of the amount diffusing, the half-times of absorption and desorption should still be equal. Very probably a nonlinear adsorption relationship would lead to the half-times being dif-

TABLE I. Summarized Data and Calculated Constants for Rates of Absorption and Desorption by Human Hair

Sorbate	Method*	pH_S	pH_D	Temp., ° C.	Q_∞ or Q_0, mM/g.	$t_{\frac{1}{2}}$, sec.	$D_{app.}$,† cm.²/sec. × 10^{11}
1 $LiBr^{82}$	D	—	—	room	—	4.0	—
2 $LiBr^{82}$	S Sub	—	—	25	8.0	1.6×10^5	—
3 $LiBr^{82}$	D Exch.	—	—	room	—	1.6×10^5	—
4 $Na^{22}Br$	D	—	—	room	—	13.7	5800
5 Na^{22} Br	S	—	—	room	2.36	1.4×10^4	4.7
6 $Na^{22}Br$	D Exch.	—	—	room	—	60	120
7 $Na_2{}^{22}SO_4$	D	6.2	5.4	22	0.072	340	—
8 $Na_2{}^{22}SO_4$	D	6.2	6.7	22	0.061	73	—
9 $Na_2{}^{22}SO_4$	S	6.2	—	20	0.080	1.7×10^4	3.1
10 $Na_2{}^{22}SO_4$	D Exch.	6.2	6.6	22	0.061	120	680
11 $Na_2S^{35}O_4$	D	6.3	5.4	25	0.070	3.0×10^4	—
12 $Na_2S^{35}O_4$	D	6.8	5.4	24	0.070	3.0×10^3	8.2
13 $Na_2S^{35}O_4$	D	6.8	6.8	24	0.070	$\sim 3.0 \times 10^3$	—
14 $Na_2S^{35}O_4$	S	—	—	25	0.074	1.0×10^4	5.8
15 $Na_2S^{35}O_4$	S	—	—	35	0.074	7.9×10^3	9.9
16 $Na_2S^{35}O_4$	S	—	—	50	0.079	2.2×10^3	27
17 $Na_2S^{35}O_4$	D Exch.	6.6	6.6	22–27	0.070	8.1×10^3	8.2
18 $Na_2S^{35}O_4$	S Exch.	6.3	—	22–27	0.066	1.1×10^4	6.7
19 $H_2S^{35}O_4$	S	2.1	—	22	0.690	1.4×10^3	37

* S = sorption; D = desorption; Exch. = exchange; Sub = sorption from 13.6 *m* solution by fibers initially at equilibrium with an 8.23 *m* solution.

† $D_{app.}$ = apparent diffusion coefficient calculated for Q/Q_∞ or $1 - Q/Q_0 = \frac{1}{2}$.

Fiber radii ranged from 2.9×10^{-3} to 3.5×10^{-3} cm.

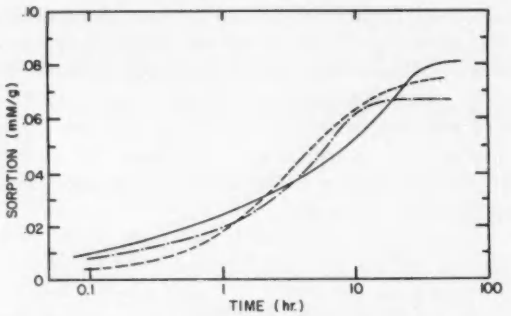

Fig. 1. Rates of absorption of components of Na_2SO_4 by hair. ——— Na^{22} absorption (0.77 *M* solution); - - - - $S^{35}O_4$ absorption (0.80 solution); —·— $S^{35}O_4$ absorption by exchange.

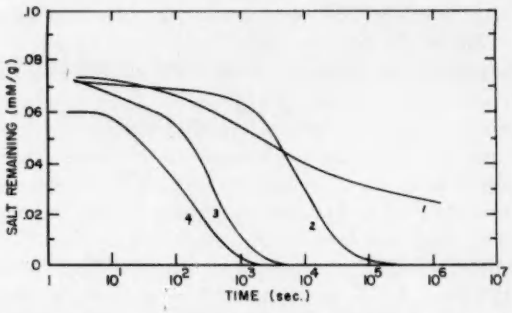

Fig. 2. Rates of desorption of components of Na_2SO_4 by hair. (1) $S^{35}O_4$ desorption into water; (2) $S^{35}O_4$ desorption by exchange; (3) Na^{22} desorption into water; (4) Na^{22} desorption by exchange.

ferent; a strong specific adsorption would lead to a ratio of half-times less than unity.

In the case of a compound absorption process, it can be shown [5] that, if the two portions of the fiber differ only with respect to diffusion coefficient, with no immobilization of diffusant or change in diffusion coefficient with concentration, the half-times should be equal. Again equivalence may well not hold for more complicated cases.

Concentration-dependent diffusion coefficients are known to cause differences between the half-times [3]. If the coefficient increases as concentration increases, desorption is slower than absorption, and if it decreases with concentration, the converse is true.

If large volume changes accompany the absorption, as is the case for the results on the uptake of alkali halides from concentrated solutions [2], a somewhat different form of concentration dependence can occur. When a water-swollen fiber is immersed in a concentrated salt solution, water diffuses out of the fiber and salt ions diffuse in. These processes may result in a measurable minimum in volume for the fiber during the absorption process [2]. During a desorption experiment, the conditions are reversed, with water diffusing into the fiber to dilute the concentrated salt solution there, and salt ions diffusing out. Since water would be expected to diffuse faster, a maximum in volume for the fiber might well be obtained, although there appears to be no evidence one way or the other. If the fiber has been swollen beyond the water-swollen condition by the salt solution, a contraction in volume would occur on desorption of the salt. Although this contraction cannot proceed any faster than the desorption, it need not proceed at the same rate. The driving forces for the contraction are essentially the same ones operative for recovery from mechanical strain; the rate is dependent on the relaxation times for the necessary polymer-chain flow processes which depend on the inherent nature of the polymer, solvent or plasticizer content, and temperature. If the swollen state is wholly or partly maintained, desorption could proceed faster than absorption. It might be noted that in cellulose the volume-recovery processes are slow enough that water can be replaced by nonswelling liquids (with a further slowing of recovery as the plasticizing action of the water is lost) so that a metastable swollen gel can be produced.

Either the mechanism just described or the existence of a diffusion coefficient which varies inversely with uptake of salt could be used to explain the large ratios of half-times of absorption and desorption observed when the interaction of hair with concentrated solutions of alkali halides is studied. The slow rate of desorption exchange for the Br^- ion may be somewhat surprising, but the equilibrium results are consonant with adsorption of the anion on sites

TABLE II. Values of $t_{1/2}/t_{1/2}$ for Various Diffusion Processes

Sorbate	Conc. treating solution	Method	pHs	pHD	$t_{1/2}/t_{1/2}$
$Na^{22}Br$	6.36 *M*	S, D	—	—	1000
$LiBr^{82}$	17 *m*	S, D	—	—	10^5
$LiBr^{82}$	14–17 *m*	Sub.-Exch.	—	—	1.0
$Na_2S^{35}O_4$	0.81 *M*	S, D	6.3	5.4	0.35
$Na_2S^{35}O_4$	0.81 *M*	S, D	6.3	6.8	3.5
$Na_2S^{35}O_4$	0.81 *M*	Exch., Exch.	6.3	6.6	1.3
$Na_2{}^{22}SO_4$	0.81 *M*	S, D	6.3	5.4	50
$Na_2{}^{22}SO_4$	0.81 *M*	S, D	6.3	6.7	230

within the fiber [1], so that this may be the cause of the slow desorption.

The one case in which desorption is slower than absorption involves the $SO_4^=$ ion, and the results are shown in Figures 1 and 2.

The various absorption curves differ in detail but are quite similar. The curve for desorption of $SO_4^=$ into water extends over more cycles of log time than would be expected for a single desorption process and indicates a very slow final process. The curves for the desorption of Na^+ and the desorption by exchange of Na^+ and $SO_4^=$ do not have this same character. Furthermore, since the fiber must remain electrically neutral, the more rapid desorption of Na^+ requires the absorption of H^+. The desorption curve for $SO_4^=$, thus, represents a composite desorption of both Na_2SO_4 and H_2SO_4. The latter process is known to be a slow process. Increase of the pH increases the rate of desorption of the $SO_4^=$ ion, presumably by lowering the H^+ concentration in the fiber.

For the double exchange process the ratio of half-times is approximately unity, as would be expected.

During absorption the cations and anions must move so as to preserve electrical neutrality. On desorption the presence of other cations in the desorption bath allows exchange processes to contribute, so that desorption can be faster than absorption. The source of the difference between the last two entries in Table II is not known, although the H^+ ion is known to diffuse relatively slowly in proteins, and the solution at pH 6.7 must contain other cations from neutralization of the acidity resulting from CO_2 in the air.

Finally the puzzling apparent sensitivity of desorption results to slight changes in the pH of the treating bath, as exemplified by items 11 and 12 in Table I, might be mentioned. The cause of this is unknown to us.

Acknowledgment

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Correction

The Absorption of a Disperse Dye by Cellulose Acetate in the Presence and Absence of Butanol, by Elizabeth P. Teulings and Howard J. White, Jr., TEXTILE RESEARCH JOURNAL **31**, 52–56 (1961).

Correction. The following omission occurs at the top of page 56, line 1, between "a" and "complex":

> "complex of smaller affinity for the fiber than the dye alone possesses. Processes which would lead to an increase in absorption would include swelling with an increase in accessibility, a decrease in the solubility of the dye in the solution, and the formation of a"

The Relation of Skein Dimensions to Strength and Yarn Number Measurements

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WHEN studying multiple-end strength testing, as it is done using the conventional 120-yd., 1.5-yd.-diameter skein, a number of variables must be considered. The length of yarn being tested has a recognized effect on results, especially when compared to single-end tests, because of the variations included within the length. The sample length not only influences the variation within but also the variation between measurements. In addition to the strength variation within and between lengths, there is also the problem of the magnitude of variation found as the test lengths and the skein perimeters are changed. These concepts have led to this study, which is designed to define the problem more thoroughly and to determine the relative magnitude of the effects involved.

Object

The object of this study was to determine the influence of both the total skein length and the skein perimeter dimensions on strength measurements and on the variation in strength and yarn number measurements.

Materials Studied

The major portion of the work was confined to combed yarns, singles and ply, with yarn numbers ranging from 12 to 96, as indicated on the charts. Some carded yarn was included to demonstrate that the principles involved were the same for both carded and combed yarns.

Testing Instruments

The effects of various skein lengths and perimeters on yarn strength were measured on an Instron console model tester. A constant crosshead traverse rate of 12 in./min. was used in conjunction with a D cell. Cell capacities ranged from 4 to 1000 lb. The load was adjusted to give as near a 50% chart deflection as possible for each set of conditions being investigated. The larger part of the study was confined to this instrument.

The Uster Dynamometer was used to measure single-strand strengths of the yarns under investigation. This instrument automatically measured and recorded the strengths of 50-cm. lengths for a predetermined number of tests. Depending upon the yarns, the applied load ranged from 200 to 2000 g. All tests were made at a setting of 5 on the variable-speed adjustment.

Variations in Test Samples

Skein lengths of 60 and 120 yd. with skein circumferences at each length of 54, 40, 30, and 20 in. were used. The same circumferences of 54, 40, 30, and 20 in. were used in preparing skeins wound with constant wraps of 80 and 40 wraps per skein.

Wraps per Skein for 1.5-Yard Perimeter

The first phase of the study was to investigate the effect of varying the wraps per skein from one to eighty while simultaneously keeping the perimeter constant at the conventional 1.5 yd., thus in effect changing total skein length while maintaining a constant perimeter.

Figures 1 and 2 show the strength levels found for the different skein lengths. The number of wraps wound were increased in increments of one from the start of one wrap to fifteen wraps; from fifteen to thirty they were increased in increments of five wraps, and from thirty to eighty in increments of ten wraps. By examining the charts, it is obvious that the relationship is linear and that the slope is a function of yarn number. Figure 3 shows the slopes for the different yarns tested; note in each case that the ply yarn slope is slightly more than twice that of the corresponding single yarn.

In addition to the expression of strength in pounds per skein, the strength in pounds per wrap was calculated by dividing the skein strength by

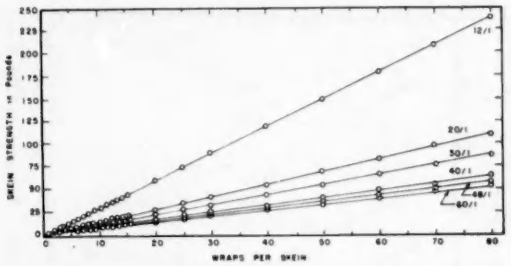

Fig. 1. Relationship between skein strength in pounds and number of wraps per skein for various combed cotton single yarns. Circumference of skein—1.5 yd.

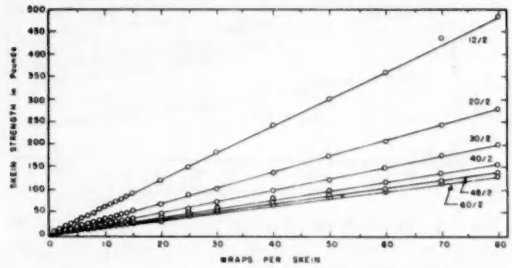

Fig. 2. Relationship between skein strength in pounds and number of wraps per skein for various combed cotton ply yarns. Circumference of skein—1.5 yd.

the number of wraps. Figure 4 shows typical examples of this relationship.

Three yarns were analyzed to see the effect of the test length on the coefficient of variation for strength. As the test length is increased this coefficient of variation decreased as would be expected from theoretical aspects. This effect is shown in Figure 5.

Constant Skein Length with Skeins of Various Perimeter

To determine the influence of skein perimeter and, thus, gauge length of testing, skeins of both 120 yd. and 60 yd. were prepared with perimeters (circumferences) of 20, 30, 40, and 54 in. Results of these measurements are shown in Figure 6 (120-yd. skein) and Figure 7 (60-yd. skein). For both the 120- and 60-yd. skeins, the 20-in. circumference gave the highest results and the 54-in. circumference gave the lowest.

To determine the influence of the number of wraps, or number of ends subjected to loading, skeins having different diameters but a constant number of wraps were prepared. Figures 8 and 9 show the effect of this difference.

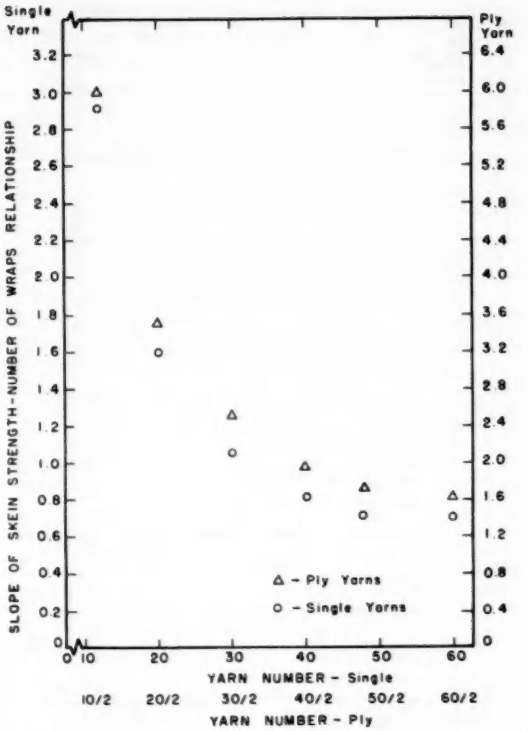
Single Yarn
Ply Yarn
SLOPE OF SKEIN STRENGTH-NUMBER OF WRAPS RELATIONSHIP
Δ - Ply Yarns
○ - Single Yarns
YARN NUMBER - Single
10/2 20/2 30/2 40/2 50/2 60/2
YARN NUMBER - Ply

Fig. 3. Relationship between yarn number and slopes for the skein strength–number of wraps relationships shown in Figures 1 and 2.

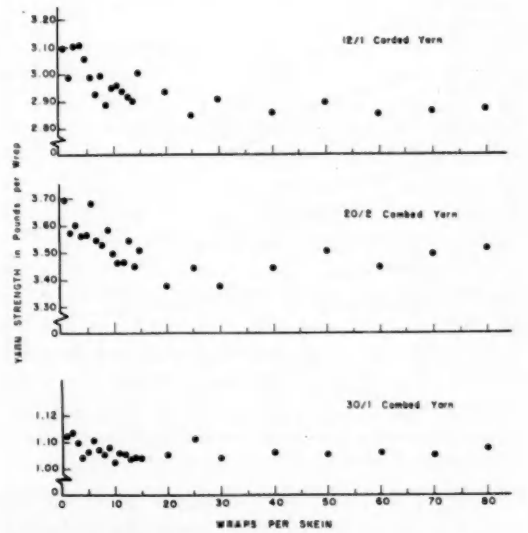

Fig. 4. Relationship of wraps per skein and pounds per wrap for cotton yarn.

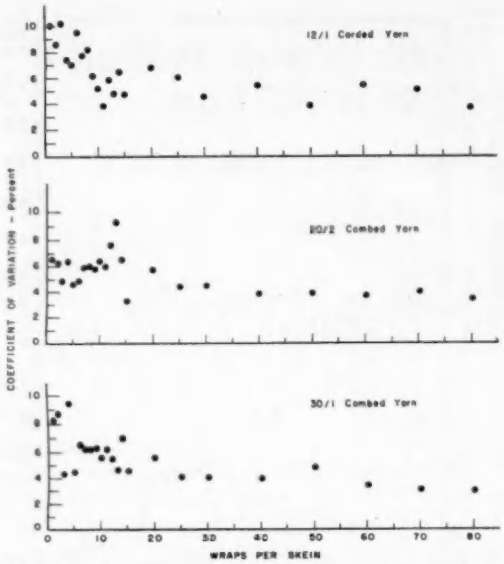

Fig. 5. Relationship between wraps per skein and the coefficient of variation of strength.

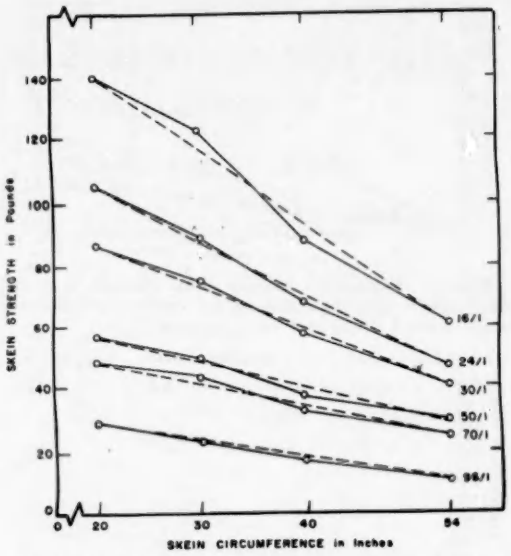

Fig. 7. Relationship between strength of a skein and skein circumference for various combed cotton single yarns. Skein length constant at 60 yd.

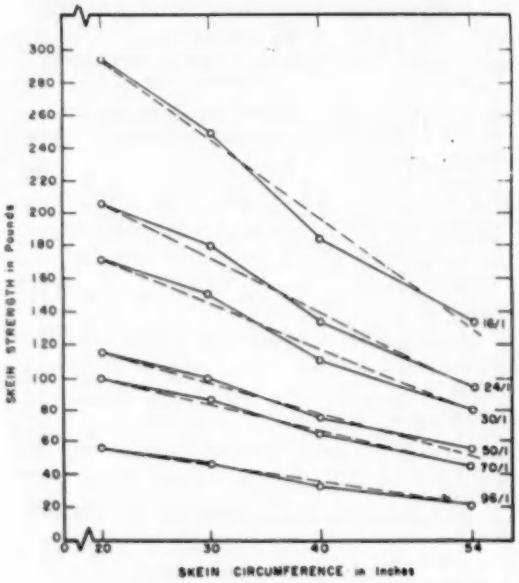

Fig. 6. Relationship between strength of a skein and skein circumference for various combed cotton single yarns. Skein length constant at 120 yd.

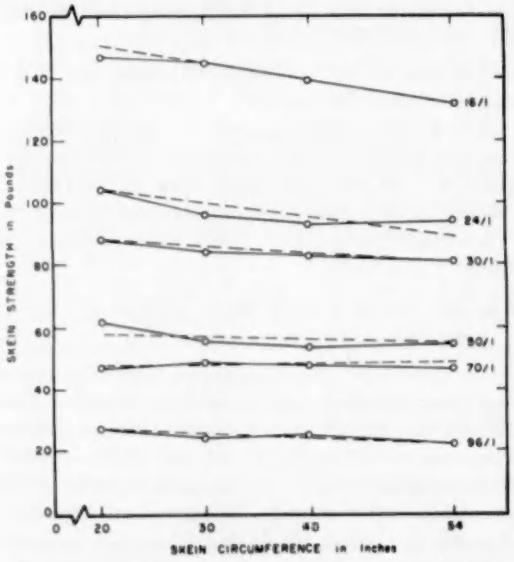

Fig. 8. Relationship between strength of a skein and skein circumference for various combed cotton single yarns. Wrap constant at 80.

In order to compare the relative effects of the various combinations of skein length and perimeter, the estimated strength per strand was calculated for each skein by dividing the skein strength by the total number of strands under test. These values as well as the single-end strength of each yarn, as measured on the Uster Dynamometer are shown in Figures 13 and 14.

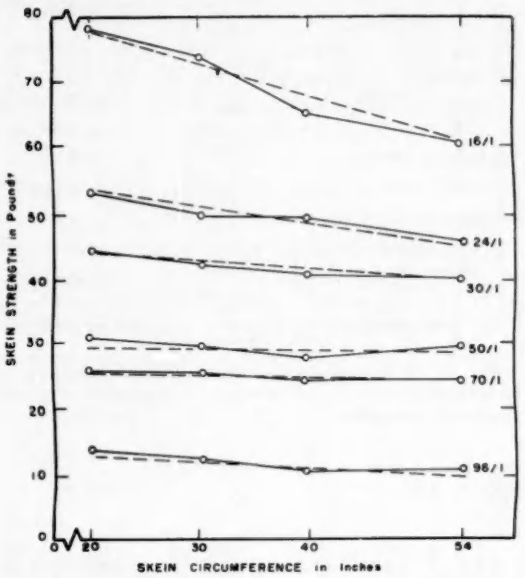

Fig. 9. Relationship between strength of a skein and skein circumference for various combed cotton single yarns. Wrap constant at 40.

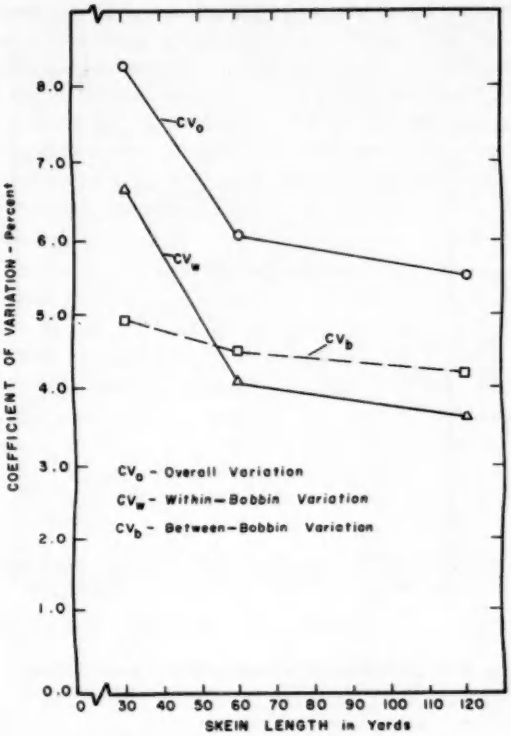

Fig. 10. Relationship between coefficient of variation of yarn strength and skein length for 1.5-yd.-perimeter skeins of 30/1 combed cotton yarn.

It is obvious that there is an optimum combination of skein length and perimeter at each length. For the 120- and 60-yd. skeins, the highest estimated single-strand strength is obtained with a 30-in. perimeter; the lowest with a 20-in. perimeter. This low estimate is most likely due to the bulk of the skein, which would tend to increase the problems of tension, tangling, and handling of the skein.

For skein tests in which the number of wraps was kept constant, the estimated single-strand strength increased as the perimeter decreased, with the rates of increase being greater for coarse yarns, so that it is apparently a function of the yarn number. This increase is most likely due to the fact that with the shorter perimeter and shorter yardage, the test more nearly simulates the single-end test. The 40-wrap, 20-in. skein gave an estimate of the single-end strength as good as or better than any other combination.

Influence of Skein Length on Source of Variations of Skein Strength and Yarn Number

The influence of skein length on the within-bobbin (CV_w), between-bobbin (CV_b), and over-all (CV_o) coefficients of variation was studied. The results shown in Figure 10 are based on four measurements per spin bobbin. As the length of the skein increased from 30 to 60 yd., there was a substantial reduction in the CV_o and CV_w. As the length was increased to 120 yd., the CV_o and CV_w continued to decrease, but at a much lower rate. The CV_b, which is a measure of the variation between bobbin averages, shows a slight tendency to decrease, but this is not as pronounced as with the CV_o and CV_w.

The influence of skein length on yarn number measurements has the same general form as in the case of strength measurements. Four skeins of different lengths were selected from spin bobbins and the yarn numbers measured. The results are shown in Figure 11. The over-all coefficient of variation, CV_o, and the coefficient of variation within bobbins, CV_w, decreased at a rapid rate as the skein length was increased from 30 to 240 yd. The CV_o represents the over-all variation of all bobbins tested, the CV_w represents the variation within the bobbins,

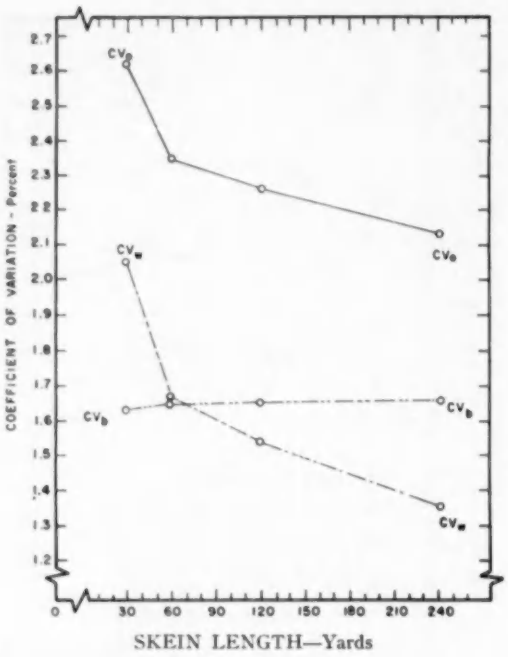

Fig. 11. Coefficients of variation of yarn number measurements for various length skeins. 30/1 combed cotton yarn.

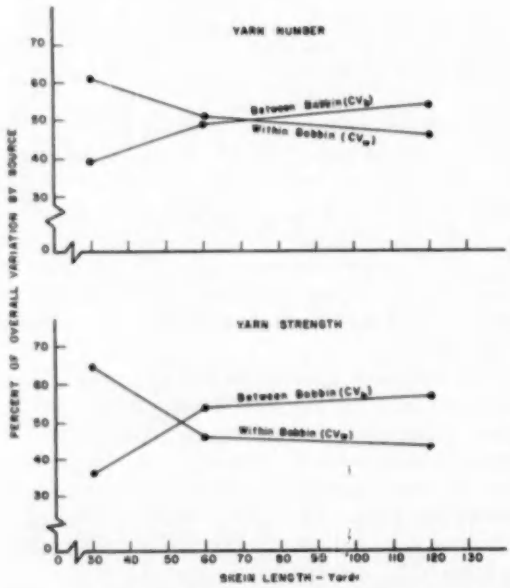

Fig. 12. Distribution of within-bobbin and between-bobbin variation, expressed as a percent of over-all variation, for different length skeins. 30/1 combed cotton yarn.

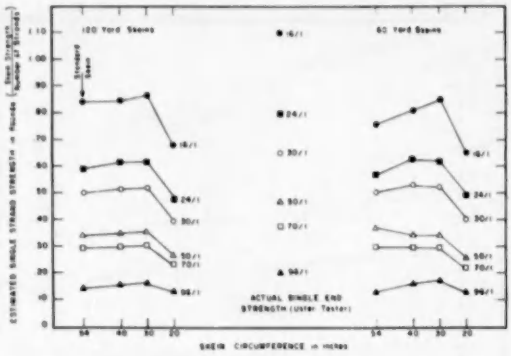

Fig. 13. Influence of skein length and skein circumference on estimated single-strand strength as compared to actual single-end strength.

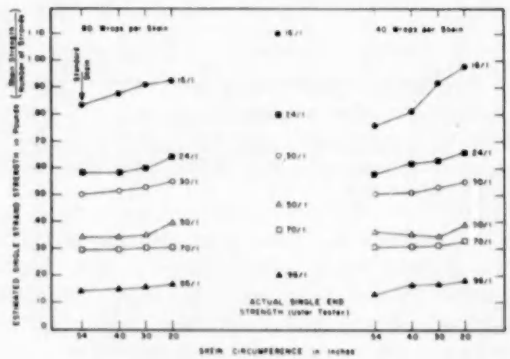

Fig. 14. Influence of wraps per skein and skein circumference on estimated single-strand strength as compared to actual single-end strength.

and the CV_b represents the variation between bobbin averages.

Figure 12 shows the influence of skein length on the distribution of within-bobbin and between-bobbin variation. For 30-yd. skeins the within-bobbin variation for yarn number represents 61% of the over-all variation and the between-bobbin variation represents 30% of the total. Since the over-all and within-bobbin variations decrease in value, as shown in Figures 10 and 11, and the between-bobbin variation remains reasonably constant, it follows that the percent of the total represented by the between-bobbin variation would increase as the length increases. It is interesting to note that the CV_w and CV_b each represent approximately 50% of the total variation in yarn number and skein strength for 60-yd. skeins.

Conclusions

As the wraps per skein increase from one to eighty, the apparent skein strength increases in a linear fashion. The slope of this relationship is a function of yarn number (Figures 1, 2, and 3).

As the wraps per skein increase, the estimated strength per wrap and its coefficient of variation decrease. The rate of decrease for both is greatest from one to twenty wraps (Figures 4 and 5).

An increase in skein circumference from 20 in. to 54 in. results in a decrease in skein strength. The rate of decrease in strength is a function of yarn number, skein length, and wraps per skein (Figures 6, 7, 8, and 9).

The over-all coefficients of variation of skein strength and yarn number decrease as the skein length increases (Figures 10 and 11). The distribution of variation within and between bobbins is also a function of skein length (Figure 12).

The 20-in. circumference skein with 40 wraps gave an estimate of single-strand strength as good as or better than any of the other lengths and skein circumferences used in this study (Figures 13 and 14).

Acknowledgments

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Tensile Behavior of Cut Skeins and Single Threads

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Abstract

A simple theoretical formulation is developed whereby it is possible to predict the load–extension behavior of an ideal bundle of textile strands in terms of the behavior of the components. Further, it is shown that the same theory can be applied in reverse to extract from the load–extension curves of the bundle the following tensile properties of single strands: (a) the extensibility distribution (b) the average breaking strength. The theory has been applied to single strands and cut skeins of cotton and viscose staple fiber yarns and is shown to be very successful in these cases.

Introduction

Starting with Peirce [6] in 1926, several workers have attempted to calculate the strength and extensibility of "ideal bundles" of strands in terms of the load–extension behavior of the component single strands. Peirce's approach has been somewhat amplified by Platt [7] and used to calculate "lea efficiencies." An elaborate statistical analysis of bundle strengths has been offered by Daniels [2]. Of late, there has been a revival of interest in this problem because of the development of the "harp tester" and similar instruments designed for simultaneous testing of collections of strands [1, 3, 4]. From the point of view of simultaneous testing, the most important question is, "Given a bundle load–extension curve, what information can we learn in reverse about the behavior of single strands?" The answer offered by the later workers so far seems to be that one can obtain the average rupture properties of single strands from a collective load–extension curve after performing some intricate and tricky analysis as well as a little guesswork. The purpose of the present paper is to show that a very simple approach can be developed for numerically calculating the load–extension behavior of an "ideal bundle" from that of the single strands, or in reverse, for calculating some single-thread tensile properties from the bundle curve. The single-thread properties in question are (a) the distribution of extensibility at break and (b) the average breaking strength. The formulation developed here has been amply

verified with five counts of cotton yarn and one of viscose staple fiber, the strands being tested in the form of singles and cut skeins (at constant rate of extension).

Part I: Theoretical Development

Theory of Synthesizing Bundle Load–Extension Curves from Singles

The numerical method. The load–extension curve of an ideal bundle can be numerically synthesized from those of the component singles without using major statistical considerations. Figure 1 shows the experimental Instron curves plotted for a number of cotton yarns. Each yarn is characterized by (a) an initial decrimping region, (b) a Hookean region, (c) a yield region, and (d) the rupture region, which may also be accompanied by a little stiffening. The coordinates at rupture of the ith thread are (e_i^*, s_i^*), the asterisk being used to denote rupture. Let N of these strands be held simultaneously and independently between the jaws of a tensile tester so that they are originally of the same length at zero tension and parallel to one another (thereby constituting an ideal bundle). When the jaws are stretched to an extension, e, the ith thread will develop a load $s_i(e)$ which can be obtained from Figure 1: The total load is the sum of the loads over the survivors, that is, over those for which e_i^* is greater than e. Thus the load–extension curve of the bundle can be obtained by summing up the loads borne by each of the threads which remain unbroken, i.e., by summing up the columns in Table I. These sums, when

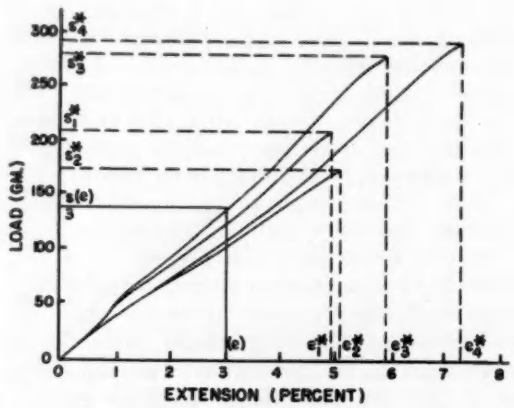

Fig. 1. Single-thread load–extension curves.

TABLE I. Synthesis of Bundle Load-Extension Curve from Single Strands

Load	e_1	e_2	e_3	e_4	—	—	—	—	—	e_n
$s_1(e)$	s_{11}	s_{12}	s_{13}	s_{14}	—	—	—	—	—	s_{1n}
$s_2(e)$	s_{21}	s_{22}	s_{23}	s_{24}	0	0	0	—	—	—
$s_3(e)$	s_{31}	s_{32}	s_{33}	s_{34}	—	—	0	0	0	0
$s_N(e)$	s_{N1}	s_{N2}	s_{N3}	s_{N4}	—	—				s_{Nn}

replotted against the values of $e_1, e_2, e_3, \cdots, e_n$, give the theoretical bundle load–extension curve of N threads, except for a certain smearing caused by the fact that each componential load–extension curve may not have been traced fully up to rupture. But by spacing the e's closely enough, this smearing can be made negligible. If the loads are divided by the linear density of N threads, one obtains the curve of nominal stress (g./tex) against nominal strain in percent. This curve should hold good for other bundles with the same or different values of N, so long as the bundle is ideal and so long as N is large enough to constitute a representative sample of the strands.

The above numerical technique was first applied to cotton single fibers and fiber bundles by Orr and Grant, but with indifferent success [5]. However, it has met with excellent success when reapplied by the present authors to cotton yarn, as will be recounted in Part II. The success of the numerical technique under a given setup indicates that the threads do not influence one another in loading (by rubbing, entanglement, etc.) so that independence and additivity of loads on single strands can be assumed.

The analytical method. When the strands in an ideal bundle behave independently and the loads on individual threads are additive, an analytical expression can be obtained for the total load developed by the bundle at each extension. Taking any thread, say the ith, its load–extension curve can be fitted with any desired degree of accuracy by the equation

$$\begin{aligned} s_i(e) &= a_i + b_i e + c_i e^2 + \cdots; && e \leq e_i^* \\ &= 0; && e > e_i^* \end{aligned} \qquad (1)$$

where e_i^* is the extension at break.

Taking threads made of cotton fiber and confining oneself to values of extension higher than the extension at yield ϵ_i, a very good approximation for the load–extension curve becomes

$$\text{Cotton: } s_i(e) = a_i + b_i e + c_i e^2; \ \epsilon_i < e \leq e_i^* \qquad (2)$$

In other words, higher powers than the square of extension may be neglected in Equation 1.

In the case of yarns of low to ordinary twist made of viscose staple fiber, even the square term is not necessary and one can write

$$\text{Viscose:}\quad s_i(e) = a_i + b_i e;\quad \epsilon_i < e \leq e_i^* \qquad (3)$$

The total load borne by the bundle is the sum of the loads borne by the individual threads. If at an extension e, $n(e)$ out of the original N threads survive, the load borne by the bundle is

$$S(e) = \sum_{n(e)} (a_i + b_i e + c_i e^2) \qquad (4)$$

provided e is larger than the maximum yield extensibility found among the n threads.

If now the parameters a_i, b_i, c_i are *uncorrelated* with the rupture extensibility e_i^*, one can rewrite Equation 4 in the form

$$S(e) = n(e)(\bar{a}_n + \bar{b}_n e + \bar{c}_n e^2) \qquad (5)$$

where $\bar{a}_n$, $\bar{b}_n$, and $\bar{c}_n$ are the average values of a_i, b_i, c_i among the n surviving threads. Thus $\bar{a}_n$, $\bar{b}_n$, $\bar{c}_n$ are statistical variates, whose mean values are

$$\bar{a} = \frac{1}{N}\sum_{i=1}^{N} a_i;\quad \bar{b} = \frac{1}{N}\sum_{i=1}^{N} b_i;\quad \bar{c} = \frac{1}{N}\sum_{i=1}^{N} c_i \qquad (6)$$

When the number of surviving threads is sufficiently large, we may replace $\bar{a}_n$, $\bar{b}_n$, $\bar{c}_n$ by $\bar{a}$, $\bar{b}$, and $\bar{c}$. Moreover, the number of survivors $n(e)$ is

$$n(e) = N\,[1 - F(e)] \qquad (7)$$

where $F(e)$ is the cumulative frequency distribution function of breaking extensibility e_i^*, i.e., the fraction of threads with breaking extensibilities less than e_i^*. Thus

$$F(e) = \int_0^e f(e_i^*)de_i^* \qquad (8)$$

and $f(e_i^*)$ is of course the frequency density function of breaking extensibility. Thus we can write ultimately the bundle load at each extension e,

$$\text{Cotton:}\quad S(e) = N(\bar{a} + \bar{b}e + \bar{c}e^2)[1 - F(e)] \qquad (9)$$

The values of $\bar{a}$, $\bar{b}$, $\bar{c}$ can be determined from the load–extension curves of a sufficiently large number of single strands; so also the distribution function of rupture extensibility $F(e)$. It can also be verified that none of the parameters a_i, b_i, c_i is correlated with the rupture extensibility e_i. Under these conditions, Equation 8 is an analytical representation of the bundle load–extension curve *in the region beyond yield strain.*

The tedium of determining the constants a_i, b_i, c_i from thread to thread can be greatly minimized as follows: Taking any load–extension curve, choose three equispaced values of extension

$$\begin{aligned} e_1 &= e_1 \\ e_2 &= e_1 + h \\ e_3 &= e_2 + h \end{aligned} \qquad (10)$$

Let the corresponding loads be s_1, s_2, s_3. If now

$$s = a + be + ce^2 \qquad (11)$$

we have

$$\begin{aligned} a &= \frac{s_1 e_2 e_3 - 2s_2 e_3 e_1 + s_3 e_1 e_2}{2h^2} \\ b &= \frac{-s_1(e_2 + e_3) + 2s_2(e_3 + e_1) - s_3(e_1 + e_2)}{2h^2} \\ c &= \frac{s_1 - 2s_2 + s_3}{2h^2} \end{aligned} \qquad (12)$$

Suitable values of e_1, e_2, e_3 for cotton yarns are 2%, 3%, 4%, giving

$$\begin{aligned} a &= 6s_1 - 8s_2 + 3s_3 \\ b &= (-7s_1 + 12s_2 - 5s_3)/2 \\ c &= (s_1 - 2s_2 + s_3)/2 \end{aligned} \qquad (13)$$

The averages of a, b, c for a sufficient number of threads (at least 50 for cotton yarn) gives $\bar{a}$, $\bar{b}$, $\bar{c}$. The fraction of survivors at any extension e is simply

$$1 - F(e) = n(e)/N \qquad (14)$$

where $n(e)$ threads have extensibility greater than e. Thus all terms in Equation 4 are known, and it can be used for numerical computation of the bundle load–extension curve.

The above treatment is an extension of the work of Peirce [6] and Platt [7]. In his pioneering work on the subject, Peirce wrote

$$s_i(e) = k_i(e) \qquad (15)$$

which is not good enough for cotton (fiber or yarn) as is clear from Figure 2.

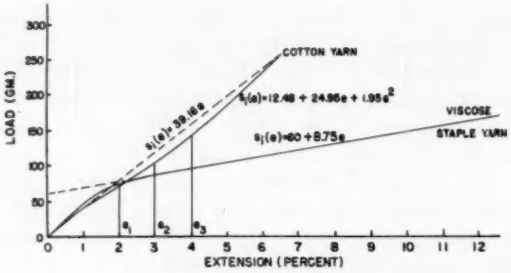

Fig. 2. Single-thread load–extension curves of cotton and viscose yarns and fitted analytical curves.

Later on, Platt considered the case

$$s_i(e) = a_i + b_i e \tag{16}$$

This representation is good enough for viscose rayon filaments or filamentous yarn, which has a high initial modulus, an early yield, and a substantially straight load–extension curve thereafter (see Figure 2). But it is inapplicable to cotton yarn, where, because of the fiber load–extension behavior as well as the yarn twist, there is a continuous stiffening with increasing extension.

On the other hand, Figure 2 shows that Equation 15 is a fairly good representation of the load–extension after the yield region of singles yarn made from viscose staple fiber. For a bundle of such threads, therefore, we would amend Equation 8 to read simply

$$\text{Viscose:} \quad S(e) = N(\bar{a} + \bar{b}e)[1 - F(e)] \tag{17}$$

The constants a_i and b_i for the individual threads can be read off directly from the y intercepts and slopes of the final parts of the load–extension curve and hence $\bar{a}$ and $\bar{b}$ determined. The bundle *breaking strength* is defined as the maximum load carried by the bundle and *extensibility at break* as the extension at maximum load. Following Peirce, these quantities can be calculated without plotting the course of the bundle load–extension curve. Rewriting Equation 4 in the form

$$s(e) = S(e)/N = (\bar{a} + \bar{b}e + \bar{c}e^2)[1 - F(e)] \tag{18}$$

the first derivative of $s(e)$ will vanish at $e = e^*$, the bundle breaking extensibility. In other words,

$$s'(e^*) = 0$$

where

$$s'(e^*) = (\bar{b} + 2\bar{c}e^*)[1 - F(e^*)] - (\bar{a} + \bar{b}e^* + \bar{c}e^{*2})f(e^*) \tag{19}$$

and $f(e^*)$ represents the frequency density function of breaking extensibility.

The value of e^* can be graphically determined by plotting the functions

$$\varphi(e) = (\bar{a} + \bar{b}e + \bar{c}e^2)f(e) \tag{20}$$

and

$$\psi(e) = (\bar{b} + 2\bar{c}e)[1 - F(e)] \tag{21}$$

against the extension e. The curves of $\varphi(e)$ and $\psi(e)$ will cross at $e = e^*$. Putting $e = e^*$ in Equation 18, the bundle breaking load $s(e^*)$ can also be determined. The same technique can be applied to Equation 16 for viscose, by modifying Equations 20 and 21 with $\bar{c}$ put equal to zero.

It must be pointed out that while this method is analytically sound, in practice it is more laborious than the mapping of the load–extension curve from Equation 8 or 18.

Theory of Analysis of Bundle Load–Extension Curve to Yield Single-Strand Properties

Having followed the development of the bundle load–extension curve in terms of the singles, a simple method of working in reverse suggests itself. Looking at a typical cut-skein load–extension curve in Figure 3, the initial steep increase in load slows down as more and more threads start breaking. If *no thread broke* during the course of the test, the load would go on increasing along a path such as $OA_1A_2A_3$ instead of along $OB_1B_2B_3$ as it actually does. The initial course of the curve (between the yield region a and the intermediate point a' at which the first thread started to break) would be given by

$$\begin{aligned} S(e) &= N(\bar{a} + \bar{b}e + \bar{c}e^2) \\ &= A + Be + Ce^2 \end{aligned} \tag{22}$$

By choosing three equispaced values e_1, e_2, e_3 of e such that e_1 is beyond the yield region and e_3 lower than the rupture extensibility of the first thread to break, it is possible by means of Equation 8 to determine A, B, C and hence

$$\bar{a} = A/N; \quad \bar{b} = B/N; \quad \bar{c} = C/N \tag{23}$$

The conditions set for the choice of e_1 and e_3 demand too much initial knowledge about the single-strand load–extension behavior. However, in practice all one has to do is to play for safety and choose a region of the initial load–extension

curve such as a_1, a_2, a_3 in Figure 3, where the load appears to increase at an increasing rate.

Having obtained A, B, C, the course of the curve along $a_1\ a_2\ a_3$ can be *extrapolated* to give the path $A_1A_2A_3A_4$. This extrapolation can be easily performed by substituting higher values of e in Equation 22 up to the maximum values encountered in the load–extension curve of the bundle as, for example, OC_1, $\cdots$, OC_4 in Figure 3, giving calculated loads OA_1, OA_2, OA_3, and OA_4. Thus the course which the bundle curve would have taken, provided no thread had been ruptured, can be plotted. As the extension increases, this will diverge more and more from the experimental curve on account of thread breakage. It will now be clear that at each extension, such as $e = OC_1$, OC_2, OC_3 the fraction of surviving threads is

$$\begin{aligned} 1 - F(OC_1) &= B_1C_1/A_1C_1 \\ 1 - F(OC_2) &= B_2C_2/A_2C_2 \\ 1 - F(OC_3) &= B_3C_3/A_3C_3 \end{aligned} \tag{24}$$

and so on. (The justification for this is simply that parameters a_i, b_i, c_i are uncorrelated with the rupture extensibility e_i^*.) It now becomes possible to estimate the function $1 - F(e)$ for various values of e. Thus *the distribution of breaking extensibility of single strands becomes known from the bundle load–extension curve.* In particular, the mean $\overline{e^*}$ and standard deviation, σ_{e^*} of single-strand extensibility can be calculated.

The rupture load on a single thread is

$$s_i(e_i^*) = a_i + b_ie_i^* + c_ie_i^{*2} \tag{25}$$

The average single thread rupture load thus becomes

$$\overline{s^*} = \bar{a} + \overline{be^*} + \overline{ce^{*2}} \tag{26}$$

or

$$\overline{s^*} = \bar{a} + \overline{be^*} + \bar{c}(\overline{e^*}^2 + \sigma_{e^*}^2) \tag{27}$$

Equation 26 also holds since a_i, b_i, c_i are not correlated with e_i^*. Since all quantities on the right-hand side of Equation 27 have been evaluated, it becomes possible to obtain the *average single-thread breaking load* from the bundle load–extension curve.

In the case of yarn from viscose staple, the same technique can be applied, but with $\bar{c}$ set equal to zero.

In summary, therefore, it is possible to obtain total information on single-thread breaking extensibility and the average single-thread breaking load from a cut-skein test, provided (1) no thread breaks below the yield strain of the thread which yields last, and (2) a parametric representation of the single-thread load–extension curve is available for each thread in which the parameters are independent of the rupture extensibility of the thread. Fortunately, all these conditions are satisfied by cotton yarn, as Part II of this paper will demonstrate. For yarns with other types of load–extension curve, it will be possible to use other types of parametric representation and to perform the same analysis provided condition (1) above is satisfied, which is usually the case. Moreover, this kind of analysis should be valid for ideal fiber bundles. But this topic is outside the scope of the present paper.

Part II: Experimental

Numerical Synthesis of Cut-Skein from Single-Thread Tensile Behavior

In order to verify the theory experimentally, load–extension curves were obtained for five sets of singles yarns made from cotton with tex values ranging from 7 to 30, as well as for yarns with tex 14 made from viscose staple fiber. The Instron tensile tester was used for strength determinations, with a nominal specimen test length of 40 cm and a rate of extension of 25%/min. The number of threads tested in each case was 150. From each

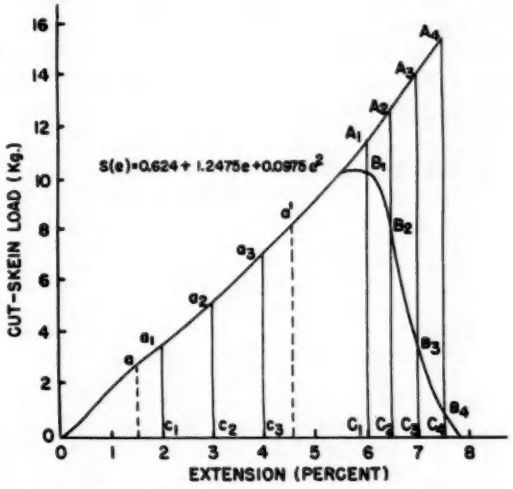

Fig. 3. Technique of extracting single-thread data from bundle curve.

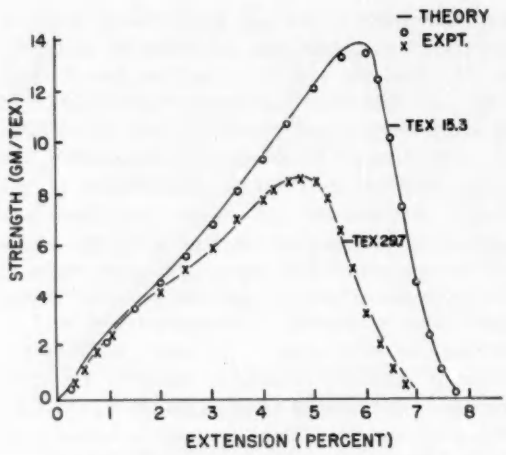

Fig. 4. Theoretical and observed stress–strain curves of cut skeins.

load–extension curve, forces were measured at intervals of 0.5% or 0.25% (Figure 1) extension. These forces were entered in Table I, thread by thread, and hence the bundle tensile curve was synthesized.

Ten skeins of 50 threads each were prepared by winding layers of yarn with the help of a small wrap reel specially made for this purpose. The density of yarn distribution was kept at 80 threads per inch per layer. To hold the individual threads in position and parallel to each other, cellulose cement and adhesive tape were applied to the skein and the cement was allowed to dry on the skein winder. The cut skein was obtained by cutting the adhesive tape in the center. The two ends were held in paper clips in order to prevent snarling and loss of twist. Each skein of 50 parallel threads was reeled out after testing 15 to 20 single threads, so as to ensure a representative sample. The cut skeins were tested on the Instron tester with a test length of 40 cm. and rate of extension of 25%/min.

TABLE II. Calculated and Observed Single-Thread Tensile Properties

Yarn tex	Strength, g		Extension, %		C. V. in extension, %	
	Calc.	Obs.	Calc.	Obs.	Calc.	Obs.
Cotton						
29.7	328.0	325.1	5.66	5.62	10.9	11.8
18.9	240.4	242.4	5.28	5.04	9.6	10.0
15.3	265.0	260.3	6.65	6.62	8.0	7.9
8.5	139.8	135.6	6.15	6.00	10.5	10.5
6.7	120.4	119.8	5.65	5.21	10.9	13.6
Viscose staple						
13.9	200.4	198.0	12.14	12.44	13.8	12.2

The load–extension curves were analyzed in the same way as for the single yarns and a mean load–extension curve was plotted as in Figure 4. It can be seen that there is hardly any difference between the theoretical and experimental bundle curves.

Analysis of Single-Thread Properties from Cut-Skein Tensile Behavior

The second aspect, namely the extraction of single-thread tensile data from the cut-skein load–extension curve, was tackled in the following steps.

Cotton Yarns:

1. The initial portion of the curve a_1 a_2 a_3 (see Figure 3) was fitted by a quadratic equation (22) and the curve representing this equation extrapolated to the highest extensions at which there were surviving threads. This curve gave the load *expected* if no threads broke.

2. The ratio of load actually observed to expected load was measured at various equispaced values of extension. The ratio gave the function $1 - F(e)$, where $F(e)$ is the fraction of threads in the sample with extensibility less than e (*vide* Equation 24).

3. The probability density function of extension at break $f(e)$ was found from $1 - F(e)$ by numerical differentiation.

4. The values of mean, standard deviation, and C. V. of breaking extension were obtained from $f(e)$ in Equation 3.

5. The average breaking load was now calculated with the help of Equations 23 and 25.

Viscose Yarn:

Here also, Steps 1 to 5 were carried out, but with obvious modifications.

The agreement between the results of this type of calculation and the observed results is shown in Table II. It will be seen that the discrepancies are well within the limits of experimental error.

The foundation of the analysis outlined above is the assumption that the parameters a_i, b_i, c_i in the quadratic representation of load–extension curves

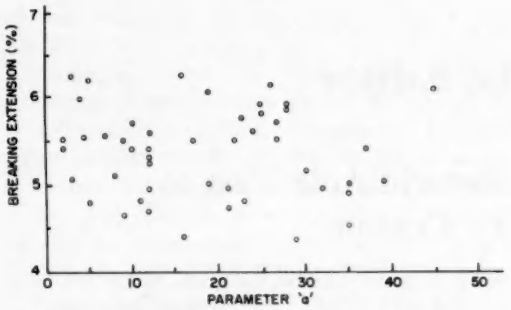

Fig. 5. Independence between breaking extension and parameters of the load–extension curve.

are distributed independently of the rupture extensibility e^*. This assumption must be verified experimentally. Figure 5 shows the parameter a_i plotted against e^* for the case of cotton yarn of 18.9 tex. The correlation coefficient is seen to be so negligibly small that it does not deserve calculation. This has been found to hold for b_i and c_i and in the case of other yarns as well.

Acknowledgment

We thank the authorities of our Association for having given permission to publish this paper.

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Manuscript received May 9, 1961.

Appendix

Illustration of Calculations on Cut-Skein Curve for Cotton Yarn of Tex 29.7

Ext. e (%)	$1 - F(e)$	Ext. e (%) mid-point	$f(e)$	$e\,f(e)$	$e^2 f(e)$
4.00	1.0000				
		4.125	0.0087	0.03589	0.14805
4.25	0.9913				
		4.375	0.0336	0.14700	0.64313
4.50	0.9577				
		4.625	0.0274	0.12673	0.58613
4.75	0.9303				
		4.875	0.0611	0.29786	1.45207
5.00	0.8692				
		5.125	0.1355	0.69444	3.55901
5.25	0.7337				
		5.375	0.1117	0.60039	3.22710
5.50	0.6220				
		5.625	0.1650	0.92813	5.22073
5.75	0.4570				
		5.875	0.1768	1.03870	6.10236
6.00	0.2802				
		6.125	0.1154	0.70683	4.32933
6.25	0.1648				
		6.375	0.0764	0.48705	3.10494
6.50	0.0884				
		6.625	0.0527	0.34914	2.31305
6.75	0.0357				
		6.875	0.0198	0.13613	0.93589
7.00	0.0159				
		7.125	0.0109	0.07766	0.55333
7.25	0.0050				
		7.375	0.0032	0.02360	0.17405
7.50	0.0018				
		7.625	0.0018	0.01373	0.10469
7.75					
			1.0000	5.66328	32.45386

Av. breaking extension (%): $\bar{e} = \Sigma e\,f(e) = 5.66\%$

Standard deviation: $\sigma_e = \sqrt{\Sigma e^2 f(e) - [\Sigma e f(e)]^2} = 0.6172$

C. V. extension $= \frac{\sigma_e}{\bar{e}} \times 100 = 10.90\%$

$A = 1.150$ $\bar{a} = A/N = 23.00$
$B = 2.459$ $\bar{b} = B/N = 49.18$
$C = 0.041$ $\bar{c} = C/N = 0.82$
$N = 50$

$\bar{s} = \bar{a} + \overline{be^*} + \overline{ce^{*2}}$
$= 23 + 49.18 \times 5.66 + 0.82 \times 32.45$
$= 328.0$ g

Letters to the Editor

Application of a Thiobarbituric Acid Test to Weathered Raw Cotton

Crops Research Division
Agricultural Research Service
U. S. Department of Agriculture
Beltsville, Maryland
June 29, 1961

To the Editor
TEXTILE RESEARCH JOURNAL

Dear Sir:

In 1959 Pan et al. [2] described a test with 2-thiobarbituric acid (TBA) to detect and measure degradation in raw cotton fiber. They found that raw cotton would react with TBA in the presence of acetic acid to produce a colored compound and that cotton severely degraded with cathode rays or by the fungus *Chaetomium globosum* produced more of this colored material in the test than did the original fiber. In continuing earlier experiments at Beltsville on the effects of preharvest weathering on cotton fiber [1], the writers were interested to determine how such weathering might influence the behavior of cotton in the TBA test.

Several series of samples of cotton fiber collected at the time of boll opening and after different intervals of weathering on the plants in the field were analyzed with TBA. The original procedure [2] was used, modified only by employment of a 100-mg. sample rather than the specified sample of 250 mg. Light absorbance after reaction with the TBA was measured on a Beckman DU spectrophotometer at 449 mμ as described [2]. The data, Table I, revealed that the weathered cotton did not display the elevated TBA values which might have been anticipated as a result of cellulosic degradation during weathering, but rather values of the same order or lower than those of unweathered samples.

TABLE I. Thiobarbituric Acid Values of Cotton Subjected to Different Periods of Preharvest Weathering *

Samples: location, crop year, etc.	TBA values* with indicated weathering periods				
	0	1 week	2 weeks	5 weeks	10 weeks
Florence, S. C., 1951	16	15	12	8	9
Florence, S. C., 1952	18	—	5	6	7
Florence, S. C., 1953	22	21	13	10	9
Florence, S. C., 1954	21	23	23	12	11
Auburn, Ala., 1954	19	13	17	9	6
State College, N. M., 1954	35	16	—	21	19
Shafter, Calif., 1952	15	—	—	—	13
Shafter, Calif., 1954	18	19	16	32	16
Florence, S. C., 1953 kier-boiled	5	4	4	4	4
Florence, S. C., 1953 water-extracted	7	7	6	9	6

* TBA value = optical density at 449 mμ ×100, employing conditions specified by Pan et al. [2] except for use of a 100-mg. sample rather than one of 250 mg.

The existence of degradation in several of the weathered samples of these series had been detected earlier by the alkali-centrifuge procedure [1] and has been confirmed by viscosity determinations of the DP (degree of polymerization) in cupriethylenediamine, using Staudinger and Daumiller's [3] equations. The latter determinations were carried out with Ostwald viscometers, with 0.03% cellulose in 0.5 *M* solvent at 20° C., and under hydrogen as the protectant gas. The DP values obtained for the unweathered samples and those weathered 10 weeks at Florence were respectively: *1951*, 2876 and 2204; *1952*, 3131 and 2579; *1953*, 3254 and 2241; *1954*, 2646 and 1719. The values for the Auburn 1954 samples, unweathered and weathered 10 weeks, were 2605 and 2227.

Boiling the samples of the Florence 1953 series in 1% NaOH for an hour prior to TBA testing, or thorough extraction with hot water, resulted in low and essentially constant TBA values, Table I. These results suggest that the compound(s) in the fiber which reacted with the TBA must be principally water-soluble. It should be pointed out that the samples from Shafter, California, Table I, had been exposed to a dry climate whereas the other samples had been weathered under humid conditions. Even cotton subjected to very long preharvest weathering did not show high TBA values. Thus, samples weathered about six months in the field

in South Carolina and Mississippi before picking had TBA values of 5.2 and 4.5.

The finding of Pan et al. [2] that cotton incubated with *Chaetomium globosum* showed increased TBA values has been confirmed. Samples of unbleached cotton duck incubated with this fungus on a glass wick–mineral salts medium for 2, 3, 4, 5, 7, and 10 days had TBA values of 3.3, 4.9, 5.3, 7.6, 10.1, and 14.4 as compared with an original TBA value of 2.6 for the unincubated fabric. These strips were thoroughly water-washed and dried after incubation and prior to the TBA test. Some caution must be taken, however, in comparing these results with those in Table I. Although the degradation occurring in some of the samples of Table I, particularly those from Florence, was undoubtedly partially microbiological in nature, the actual degree of degradation was much greater in the incubated ravelled strips. The strips showed strength losses by the 6-in. ravelled-strip method of 28%, 38%, 45%, 60%, 91%, and 100%, respectively, whereas field-weathered samples of the nature of those in Table I rarely show a Pressley strength loss or a yarn strength loss of over 10% of the unweathered value for a 10-week weathering period [1].

The present communication is not offered as a criticism of the earlier paper on TBA [2] but rather as a supplementary note relating to the application of the method to fiber degraded in the field under conditions other than those of immediate concern to the originators of the procedure.

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G. K. HARRISON
P. B. MARSH

Increase of Instron Sensitivity as an Aid to the Study of the Mechanical Properties of Cotton Fibers[1]

Institute for Fibres and
Forest Products Research
Jerusalem, Israel
August 7, 1961

To the Editor
TEXTILE RESEARCH JOURNAL

Dear Sir:

During the course of our work on the crimp of cotton fibers it has become necessary to observe the behavior of the fibers in the load range of 0–100 mg., the range which corresponds to the uncrimping of the fibers.

The Instron tester[2] is equipped with a load-weighing system providing a maximum full-scale sensitivity of 2 g.; this sensitivity is obtained when using the "A" cell and the "High" position. As this was not sufficient for our purposes, we found it necessary to increase the sensitivity of the weighing system in order to facilitate the stress–strain measurements of single cotton fibers in the desired low range of loads.

At first we attempted to do this by changing the value of the load resistors in the discriminating rectifier circuit. By this method we obtained a full-scale sensitivity of 200 mg. However, we had to abandon this arrangement because the zero adjustment became very unstable. The zero point drifted as much as ± 1% of full scale. The same instability of the zero adjustment was noted when we increased the sensitivity by reducing the voltage on the slide wire. We were later notified by the Instron Corporation that such a modification had been tried by other users of the Instron tester, but that they had encountered the same problems.

Next we increased the sensitivity by increasing the gain of the load amplifier itself. We did this by reducing the amount of feedback voltage in the first two stages as well as in the last two stages. This resulted in a maximum full-scale sensitivity of 200 mg. There was still a noticeable wiggle of the pen,

[1] This work forms part of the research being carried out at this Institute under grant No. FG-Is-103-60 issued by the Agricultural Research Service, United States Department of Agriculture.
[2] Instron Engineering Corporation, Canton, Mass.

which usually was not more than 1% of full scale, but at times reached a value as high as 1.5%. We therefore restored the feedback network in the first two stages to its original value. We now get a maximum full-scale sensitivity of 300 mg. The wiggle of the pen is approximately ± 1 mg., or a total of 0.7% of full scale. Due to the high sensitivity of the modified load-weighing system, the adjustment of the balance potentiometer is now very critical. The inherent instability of the pen position in "High" becomes even more pronounced now that the sensitivity is so much greater. There is therefore a noticeable drift of the pen if a test is performed over a longer period of time. However, over a period of 3–5 min. the position of the pen remains stable. This is satisfactory to us, as the particular experiments on each fiber usually take approximately 1 min.

In conclusion it may be said that this modification of the load-weighing system may be used whenever tests are performed over a short period of time and where the wiggle of the pen is not a disturbing factor.

D. GOLDSCHMIDT
M. SHILOH

Crystalline Orientation of Roselle Fiber

Technological Research Laboratories
Indian Central Jute Committee
Regent Park, Calcutta-40, India
July 14, 1961

To the Editor,
TEXTILE RESEARCH JOURNAL

Dear Sir:

Roselle fiber (*Hibiscus sabdariffa*) is an important substitute for jute and is mostly used for blending with the latter fiber. Charterjee et al. [3] and Das Gupta [4] studied the chemical properties, and Bandopadhyay et al. [1] and Bose [2] some physical properties of that fiber.

The present work was undertaken in order to study the crystalline orientation of roselle fiber and also to investigate variation in orientation in raw, delignified fibers and in fibers treated with different strengths of caustic soda. The Hermans' orientation factor, f_x, the average angle of orientation, α_m, and the angle for 40% intensity were determined.

Samples of alkali-treated fibers were prepared by treating defatted roselle fiber with different strengths of NaOH solution for 2 hr. and subsequently washing them thoroughly in water and drying them in air without tension. Delignified samples were prepared in the usual way by the Textone process. X-ray diffraction photographs, suitable for microphotometer scanning, were taken with Cu-Kα radiation (Ni-filtered) from a Philips' PW 1009 sealed tube working at 35 kv and 20 ma. The camera used was a flat casette plate camera.

Following Hermans et al. [6, 7] a series of micro-

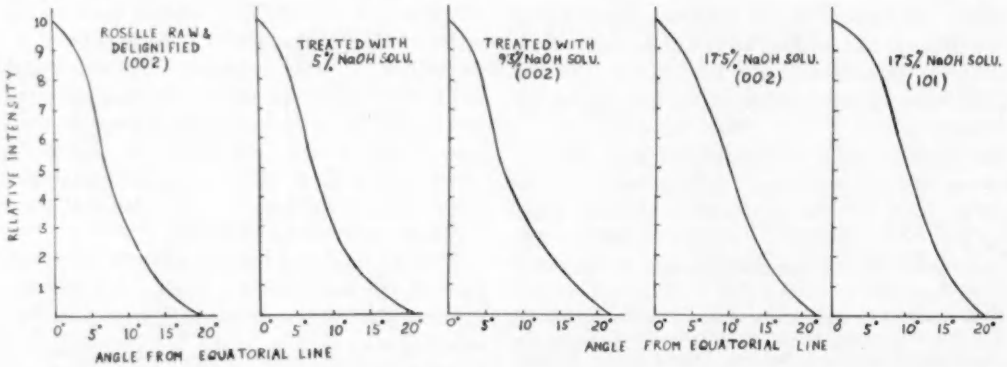
RELATIVE INTENSITY
ROSELLE RAW & DELIGNIFIED (002)
TREATED WITH 5% NaOH SOLU.
TREATED WITH 9% NaOH SOLU. (002)
17.5% NaOH SOLU. (002)
17.5% NaOH SOLU. (101)
ANGLE FROM EQUATORIAL LINE
ANGLE FROM EQUATORIAL LINE

Fig. 1. Relative intensity against angular distances from the equatorial line along the Debye–Scherror arcs of diffraction spots.

photometer records of (002) and composite (101) and ($10\bar{1}$) interference were made, starting from the equatorial lines of the diffraction photographs and proceeding along radial lines at angular intervals of 2° 30′. In the case of raw fiber, it has been found that intensity distribution along the arcs of (101) and (002) are the same, hence only intensity distribution for (002) is shown in the first three parts of Figure 1. Only in the case of fibers treated with 17.5% NaOH solution were both (002) and (101) reflections taken into consideration for calculating α_m and f_x.

The values of f_x, α_m, and the angle for 40% intensity in the intensity distribution curves for (002) reflections are given in Table I. The results obtained show that the average angle of orientation α_m and f_x change when the fiber is treated with 9.3% NaOH solution, and at mercerizing strength (17.5%), the values change further.

The average angle of orientation α_m and orientation factor f_x for raw and delignified roselle are found to be $\alpha_m = 9° 12'$ and $f_x = 0.962$, which correspond to the values for lowest grade of jute as found by Sen and Chowdhury [9]. In the case of ramie [6] these values are 7° 36′ and 0.973, and for mesta [8] (another important substitute for jute) they are 11° 36′ and 0.940, respectively. It is also found that at mercerizing strength, f_x decreases from 0.962 to 0.942, whereas the value for ramie [6] decreases from 0.973 to 0.900, for jute [5] from 0.96 to 0.79, and for mesta [8] from 0.940 to 0.905.

TABLE I.

Sample	40% Intensity angle	Hermans' average angle of orientation	Orientation factor	
Raw	8° 20′	9° 12′	.962	
Delignified	8° 20′	9° 12′	.962	
Treated with 5% NaOH soln.	8° 24′	9° 23′	.960	
9.3% NaOH soln.	8° 42′	10° 18′	.952	
17.5% NaOH soln.	10° 30′ (002)	11° 32′ (002)	.940	.942
	11° 30′ (101)	11° 3′ (101)	.945	

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R. K. BASU

Method of Measuring Torsional Rigidity of Fibers at Elevated Temperatures and Pressures

Southern Regional Research Laboratory [1]
New Orleans 19, Louisiana

July 21, 1961

To the Editor
TEXTILE RESEARCH JOURNAL

Dear Sir:

The effects of temperature and solvents on the shear properties of polymer fibers are easily followed from changes in their torsional rigidity [2, 3, 4]. The remarkable simplicity of the torsion pendulum belies the usefulness of the method. This nondestructive method is applicable to measurements of torsional rigidity and internal friction in the regions of second-order transitions, especially to determine the effects of solvent vapors at elevated temperatures and pressures. In this report are given a brief description of the torsion pendulum with a conditioning chamber and the data for several chemically modified cotton fibers. The results illustrate the potentialities of the method for analyzing the effects of heat and solvent vapors under pressures of several atmospheres on the torsional behavior of single cotton fibers. Similar analysis of tensile stress–strain properties requires more elaborate equipment.

[1] One of the laboratories of the Southern Utilization Research and Development Division, Agricultural Research Service, U. S. Department of Agriculture.

Apparatus

A sketch of the longitudinal cross section of the apparatus is shown in Figure 1. The chamber (A) is made from a Pyrex tube 2.5 cm. in diameter with walls 2 mm. thick. The chamber is closed with a ball and socket joint, lubricated and clamped to withstand pressures both above and below those of atmospheric conditions. Special safety precautions must be exercised when the apparatus is used at extreme pressures. The Pyrex tube fits inside a heating jacket (B), which is a metal tube wrapped with glass cloth, nichrome resistance wire, and insulation of about 6 mm. thickness. At the level of the pendulum, two windows (E) opposite each other in the jacket allow observation of the oscillations. The temperature is controlled with a variable transformer connected to the resistance wire. Temperature is measured with a thermocouple placed near the fiber. Vapor pressures are controlled by using appropriate drying agents or water in the bottom of the chamber.

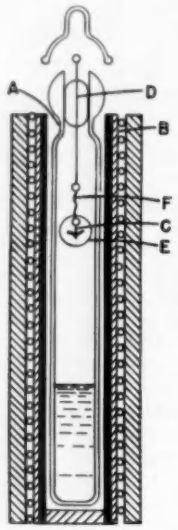

Fig. 1. Cross-sectional view of the apparatus for measuring the torsional rigidity of cotton fibers. A, pyrex glass chamber; B, heating jacket; C, pendulum bob; D, pendulum holder; E, window; F, fiber.

The pendulum bob (C) is V-shaped to minimize effects of condensation. Droplets which concentrate directly beneath the specimen have minimum effects on the moment of inertia and calculations of pendulum moments. Attached to one arm of the pendulum bob is about 3 mg. of transformer iron, which is counterbalanced on the other arm with copper. While moments of odd-shaped pendulum bobs are difficult to calculate directly, they can be obtained by comparison, using a long filament with a simple pendulum of known moment.

The single fiber is attached to the sample holder (D) and the pendulum bob (C) using a glue that withstands the conditions of the test. The sample holder with specimen and bob attached is shown in Figure 1 as positioned in the chamber.

After the temperature and pressure are brought to the desired conditions, a strong magnet is passed near the apparatus to induce rotary oscillation and then placed at an appropriate predetermined position to neutralize the horizontal component of the earth's magnetic field. When the amplitude of swing is such as to produce about 1% maximum shear strain in the fiber the period of oscillation is determined. Changes in internal friction or loss factor can be estimated from the rate of decay of the amplitude of the pendulum's swing.

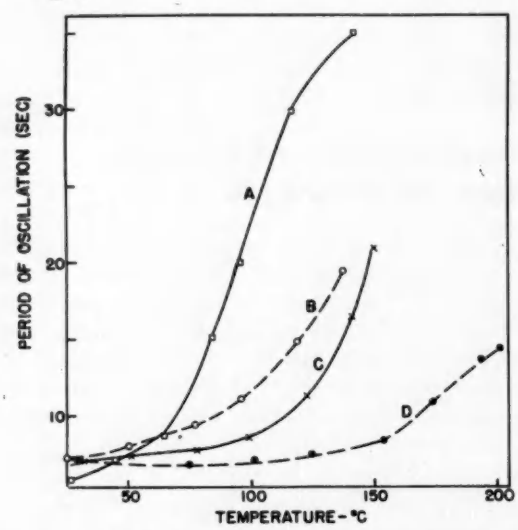

Fig. 2. The effects of temperature and water vapor on the period of oscillation of torsion pendulum. A, benzylated cotton fiber in saturated water vapor; B, benzylated cotton fiber in very dry air; C, fully acetylated cotton fiber in saturated water vapor; D, fully acetylated cotton fiber in very dry air.

Results

The effects of temperature on torsional rigidity as determined with the apparatus have been reported for benzylated cottons of several degrees of substitution and for acrylic fibers [1]. To illustrate the effects of heat and water vapor on torsional rigidity, curves are given in Figure 2 for the periods of oscillation at different temperatures of partially benzy-

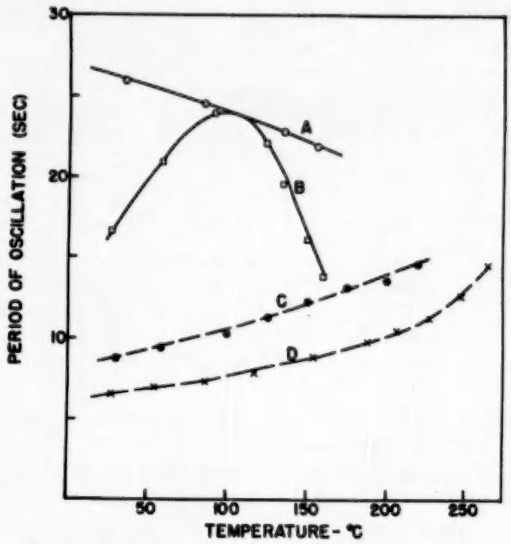

Fig. 3. The effects of temperature and water vapor on the period of oscillation of a torsion pendulum. A, untreated cotton fiber in saturated water vapor; B, mercerized cotton fiber in saturated water vapor; C, untreated cotton fiber in very dry air; D, mercerized cotton fiber in very dry air.

lated and fully acetylated cotton fibers. The results under dry conditions are shown in Curves B and D and in saturated water vapor up to 150° C. (70 lb./in.^{2} steam) in Curves A and C. The dry and saturated conditions were attained by placing anhydrous calcium sulfate or water, respectively, in the bottom of the chamber.

The rapid increase in period of oscillation with increase in temperature indicates a rapid decrease in shear modulus. Corrections for the increase in fiber cross-sectional area caused by swelling would reduce further the shear modulus value. Shear modulus values could not be calculated accurately because of the difficulties in estimating the diameter and shape, especially in the case of swollen fibers. The effect of water vapor is especially pronounced above 100° C. Much less solvent effect was noted in steam at atmospheric pressure, particularly above 100° C.

Figure 3 gives the curves for untreated and mercerized cotton fibers. The decrease in oscillation period when temperature of the untreated cotton in saturated water vapor is increased (Curve A) is attributed to the lower moisture absorption capacity of cotton at the higher temperature, thereby increasing the shear modulus. Curve B, for mercerized cotton fibers under similar conditions, shows a maximum near 100° C. Fibers in a very dry atmosphere, Curves C and D, show decreases in modulus as the temperature is increased. A complete analysis of the influence of atmospheric conditions on the mechanical behavior of fibers has not been attempted. However, the method is adaptable to investigations of mechanical behavior under atmospheric conditions which are difficult to achieve in tension and recovery measurements.

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ROLLIN S. ORR
JAMES N. GRANT

Cross-Linking Cotton with Dimethylol Urea and Alkaline Catalyst

Southern Regional Research Laboratory[1]
New Orleans 19, Louisiana
July 14, 1961

To the Editor
TEXTILE RESEARCH JOURNAL

Dear Sir:

Methylol amides, such as urea-formaldehyde condensates, are widely used as finishing agents for cotton to produce wrinkle-resistant and wash-and-wear fabrics. These agents are usually applied with acid catalysts and, under these conditions, are widely believed to produce their effects by cross-linking the cellulose. Methylol ureas, however, also impart similar properties to cotton when applied with alka-

[1] One of the laboratories of the Southern Utilization Research and Development Division, Agricultural Research Service, U. S. Department of Agriculture.

TABLE I. Properties of Cotton Print Cloth Treated with Dimethylol Urea

Treating bath composition		Fabric analysis			Fabric properties		
DMU, %	Catalyst	N, %	CH_2O, %	CH_2O/Urea	Crease recovery angle (Monsanto) (W + F), deg.	Tearing strength (Elmendorf) (W), g.	Moisture regain, %
4	Na_2CO_3 (1.0%)	0.62	0.99	1.49	259	687	6.0
4	$Zn(NO_3)_2 \cdot 6H_2O$ (0.3%)	0.61	0.93	1.42	262	740	5.9
8	Na_2CO_3 (1.0%)	1.15	1.96	1.59	286	620	5.1
8	$Zn(NO_3)_2 \cdot 6H_2O$ (0.6%)	1.15	1.87	1.52	289	667	5.5

line catalysts. It is maintained that different reactions occur under alkaline conditions. Gagliardi [1] and, more recently, Steele [4] and Mehta and Mehta [3] have published data to show the differences in the properties imparted to fabric under the two types of catalytic conditions. These authors exclude the possibility of cellulose etherification under alkaline conditions.

In Table I are shown the results obtained in this laboratory by treating cotton fabric with dimethylol urea at two different concentrations using an acidic catalyst, zinc nitrate, and an alkaline catalyst, sodium carbonate. These treatments were applied by the usual padding operation followed by drying at 60° C. for seven min. and curing at 160° C. for three min. Following the cure, the fabrics were given an alkaline afterwash. The data show that at either level of treatment, there were no significant differences between the fabrics treated under the two catalytic conditions.

The similarity of properties of the fabrics treated under the two conditions suggests that the same reactions occur in both instances. If cellulose cross-linking is the effective reaction, it is necessary that dimethylol urea etherify cellulose under alkaline conditions. Etherification of simple alcohols by dimethylol urea is claimed in the patent literature, but the products described are poorly defined and of doubtful composition. To demonstrate that etherification with dimethylol urea can proceed under alkaline conditions, the following methylation of dimethylol urea with potassium carbonate as catalyst was performed.

Dimethylol urea was prepared by dissolving 18 g. of urea (0.3 mole) in 50 g. of 36% formalin (0.6 mole formaldehyde) made slightly alkaline with sodium carbonate. The dimethylol urea that precipitated on standing at room temperature was filtered and dried under vacuum at 35° C.

The prepared dimethylol urea, 18 g. (0.15 mole), was mixed with 100 g. of methanol and 21 g. of anhydrous potassium carbonate. This quantity of carbonate served as catalyst and dehydrating agent to remove water produced in the etherification. After refluxing for 1 hr., the mixture was filtered, the filtrate carefully neutralized with oxalic acid, and refiltered. The filtrate was concentrated under vacuum, below 40° C., to about 40 g. After concentration the filtrate was still neutral. The concentrate was warmed slightly to give a clear solution and then chilled to precipitate 5 g. of product, m. p. 100–101° C. Reported melting point of bis(methoxymethyl)urea is 101° C. [2]. Analysis of the product showed 18.72% nitrogen and 39.32 formaldehyde; theoretical composition of bis(methoxymethyl)urea is 18.91% nitrogen and 40.53% formaldehyde. Further concentration of the filtrate yielded 4 g. more of product, m. p. 97–99° C.

In another preparation the same procedure was used with the exception that the solution was not neutralized before isolation of the product. Because of the difficulties in isolation of a pure product from the alkaline solution, only a low yield of bis(methoxymethyl)urea was obtained.

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J. G. FRICK, JR.

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